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MODEL STUDIES FOR A FRIABLE SANDSTONE

by

MERVYN FRANCIS MILLIGAN




A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND
RESEARCH IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR
THE DEGREE OF MASTER OF SCIENCE

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled "MODEL STUDIES FOR A FRIABLE SANDSTONE" submitted by Mervyn Francis Milligan in partial fulfilment of the requirements for the Degree of Master of Science in Civil Engineering.

To Geraldine

ABSTRACT

This thesis investigates the geotechnical aspects of the structure inherent in some friable sandstones, of which the McMurray Sandstone is one. Due to the interpenetrative nature of the sand grains, high angles of internal friction are achieved at low effective normal stresses. The overall effect of this structure is to produce a Mohr failure envelope with extreme curvature.

It is shown that diagenetic alteration of the sediment after deposition causes this particular structure to develop. The diagenetic mechanisms responsible for this include pressure solution and/or authigenic quartz overgrowths.

Methods are used to simulate this structure in the laboratory. Because of the low reactivity of quartz, a model material is developed using glass beads and hydrofluoric acid. Photographic analyses of the beads after dissolution indicate that the original tangential contacts have been destroyed, and replaced by concavo-convex or long contacts. Shear results show curvature of the Mohr failure envelope when the latter contacts prevail.

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CHAPTER I

INTRODUCTION

In classical soil mechanics concepts for granular materials, a linear Mohr failure envelope is usually assumed. The inclination of the failure envelope depends on many factors, among these mineral characteristics, particle size and shape and roughness of grains are important. However, when dealing with a diagenetically altered material as the McMurray Sandstone, other factors have to be taken into consideration.

This sandstone shows an unusual structure. The grains are interlocked in a ball and socket configuration. The absence of cement is pronounced. The interlocked structure results in shear characteristics never before recognized in geotechnical engineering.

Very high angles of internal friction, approximately 65 degrees, are observed at low effective normal stresses. The process of shear is accompanied by high dilatancy at these stresses. At higher effective normal stresses, angles of internal friction of approximately 35 degrees are obtained. This is due to the roughness of the grains. Reasons for this include solution and/or the formation of secondary quartz on the sand grains after deposition. These diagenetic processes result in an interlocking structure and impart a pseudo-angularity to the sand grains.

The purposes of this thesis are to investigate the diagenetic mechanisms which brought about this particular structure in the sand grains. A model to simulate this structure, and upon which subsequent shear tests may be carried out, is also to be examined. Contents of the various chapters of the thesis are summarized below.

Chapter II presents the geotechnical properties of the McMurray Sandstone. As well as shear strength data, thin sections and electron microscopy of the natural material are examined.

The processes dealing with the diagenetic alteration of the sand grains are discussed in Chapter III. A correlation is made with the St. Peter Sandstone on the basis of structure and strength data.

In Chapter IV the experimental model used to simulate the structure of the McMurray Sandstone is presented and discussed.

The results from the experimental tests are given and discussed in Chapter V. Thin sections and electron microscopy of the test samples are also presented. Correlation is made with the behaviour of randomly jointed rock.

Chapter VI summarizes the conclusions drawn from this investigation.

In this thesis, scientific metric units were used rather than SI units ($1\text{kg}/\text{cm}^2 = 98.1\text{ kPa}$).

Chapter II

MCMURRAY OIL SANDS - STRENGTH PHENOMENON

2.1 Introduction

This chapter presents some of the geotechnical properties of the oil rich sandstones comprising the Athabasca Tar Sands. First however, some insight into the geology of the region is given. Location and stratigraphy of the area are presented ending with a detailed description of the McMurray Sandstone.

The next section discusses some of the postulates of strength that have been made concerning the McMurray. These are found to be erroneous or not entirely applicable. Strength data, in the form of shear box tests on the natural material are then examined. These give results not before encountered in classical soil mechanics.

Finally, thin sections made from the undisturbed material and scanning electron photomicrographs of the individual grains are inspected.

It is perhaps relevant to distinguish what is meant by a sandstone and a sand, in the context of the McMurray. To the geologist, the McMurray represents a semi-lithified material and is referred to as a sandstone. However, as far as the geotechnical engineer is concerned, the McMurray represents a sand, that is, a material without cohesion.

In this thesis, the author will use both terms, but it is recognized that the latter is more appropriate.

2.2 Athabasca Tar Sands

2.2.1 Location

The Athabasca tar sands are located in northeastern Alberta, close to the valley of the Athabasca River. They are situated 483 kilometres northeast of Edmonton, Figure (2.1.).

The tar sands underlie an area greater than 26,000 square kilometres and crop out extensively in the valleys of the Athabasca River and its tributaries. Estimates of the heavy oil contained in the Athabasca tar sands are in excess of 700 billion bbl (Pow et al., 1963).

2.2.2 Stratigraphy

The Athabasca tar sands were deposited in Cretaceous time. Deposition took place on Devonian limestones adjacent to the Precambrian shield in northeastern Alberta.

The sequence of strata unconformably overlying the Devonian rocks may be divided into three major stratigraphic units, which are, in descending order, (Carrigy, 1971):

Grand Rapids Formation (115 metres thick), consisting of thick, fine to medium-grained feldspathic sandstone beds separated by intervals of laminated glauconitic siltstone and silty shale with thin coaly beds in the upper part;

Clearwater Formation (80 metres thick), consisting of marine dark grey fossiliferous silty shale and laminated siltstone, with fine-grained glauconitic cherty siltstone at base and in upper part, gradational with overlying Grand Rapids Formation;

McMurray Formation (0-100 metres thick), consisting of fresh to brackish water lenticular sandstone and laminated grey siltstone with thin interbeds of dark grey foraminiferous bearing shale in the upper part, disconformable with the Devonian limestones, conformable with the overlying Clearwater Formation.

The irregular pre-Cretaceous topography, developed on the Devonian surface, plays a major role in controlling the thickness and areal extent of the overlying McMurray Formation. The erosional Devonian surface consists of a series of alternating valleys and ridges. The McMurray sands and silts therefore tend fill in these Devonian limestone valleys, and thin over the ridges. This general levelling out effect by the McMurray Formation is somewhat obscured by the local post-Cretaceous folding (Stewart, 1963). Furthermore, the distribution of oil contained in the McMurray Formation is, in part, controlled by this ancient topography.

The Cretaceous strata dip uniformly southwest toward the foothills of the Rocky Mountains at a rate of 1.0 metre/kilometre. However, local exceptions exist. These are

caused by the post-Cretaceous collapse of Devonian limestones due to salt removal from the underlying strata. In fact, vast reserves of salt underlie the McMurray area with tonnages comparable to the estimated oil reserves (Carrigy, 1959). Extensive outcrops of a pure limestone (95 percent calcium carbonate), are also present in the area.

A rough three fold division of the McMurray Formation can be recognized, comprising the Upper, Middle and Lower Members (Carrigy, 1959). The Upper Member essentially consists of fine-grained, oil rich quartz sands, horizontally bedded. The Middle Member consists of medium-grained oil rich sands, interbedded with lenticular siltstone. Shale, coal and ironstone beds are also present as well as vegetable remains and pyrite nodules. In the Lower Member, basal clays and shales are present in the coarse-grained oil rich sands. Some wood, lignite and coal may also be found.

2.2.3 McMurray Sandstone

The McMurray Sandstone is a friable sandstone averaging about 60 metres in thickness. It is early Cretaceous in age and outcrops along the lower Athabasca River. Deposition took place in a lacustrine to delta environment.

The McMurray contains the majority of the oil in the Athabasca reserves. Oil infiltration is not complete everywhere however, and oil-impregnated sands may be

observed overlying clean white sands at some outcrops. The whiteness is due to the high quartz content.

Carrigy (1971) illustrates the significant features of the McMurray Formation in terms of grain size, clay content, oil content and by examination of the self potential and resistivity curves for the material, Figure (2.2).

The grain size data indicate a fining upward profile coupled with an increase in the clay size material. Both these properties are reflected in the self potential curve. A maximum negative deflection is recorded at the base with a gradual return to the shale line (Clearwater Formation) at the top of the unit.

The oil content also is greater at the base, gradually decreasing toward the top. The oil saturation is picked up by the resistivity log, which traces a line showing maximum deflection at the base and a gradual return to the base line at the top.

A typical oil-sand slope, illustrating these features is shown in Figure (2.3).

Carrigy (1966) shows that the coarse-grained sands are well graded whereas the fine-grained sands are poorly graded. Individual grains have a greater tendency to be angular than rounded. Chemical analysis of the sand gives a quartz content greater than 95 percent.

Other minerals present include feldspars and mica. Clay minerals are present in minor amounts (about 2 percent), and consist of illite and kaolinite. Montmorillonite is very rare or entirely absent. Heavy minerals present include garnet, zircon and apatite.

The natural oil sand is water wet with bitumen and small amounts of gas occupying the interstices, Figure (2.4). The maximum amount of bitumen, 18-19 percent by weight, is present in the well sorted, clean coarse sands. The variation in oil content can be explained by concomitant variation in texture of the sediment.

2.3 Strength Phenomena

The high strength of the oil sands is demonstrated by the steep valley walls, Figure (2.3). Slope angles of 65 degrees may be found in the fine-grained oil sands along the banks of the Athabasca River. The angle of repose for fine-grained sand usually lies between 28-34 degrees. Thus the high strengths of the oil sand may be appreciated.

This anomaly poses the question of origin of strength. Postulates of this origin include, (Dusseault and Morgenstern, 1975):

(a), cohesion by bitumen ; assuming the bitumen to be an Newtonian fluid, as proposed by Bowman (1967), it may be shown that no appreciable gain in strength results from the interaction of the bitumen. Also, bitumen being a viscous liquid, it will not contribute to long term strength.

(b), high negative pore fluid stress acting ; shear box testing of undisturbed material indicates that some enhancement of strength may result from this. However, whether the same effects are produced in situ, cannot be verified at this time.

(c), physical cementation of grains ; on the surface outcrops of the McMurray, physical cementation is uncommon (Dusseault, 1975). Some ironstone concretions along with iron oxide cementation do exist at certain localities, but overall, cementation is rare. Quartz and calcite cements may also be observed in some places.

(d), complex interfacial tensions in the quartz-bitumen-water system; the interaction of the quartz, bitumen and water system is not fully understood. It is assumed however, that such interaction would contribute little to strength.

As noted before, the sand grains are water wet with bitumen and gas occupying the interstices. At the surface of an outcrop therefore, the water will evaporate leaving the quartz grains bitumen wet. The bitumen, acting as a hydrophobic agent, would prevent the ingress of meteoric water at the slope surfaces.

Another result of this would be a reduction in the freeze thaw process. This is commonly observed where fine-grained oil-impregnated sands overlie clean sands. The latter invariably shows angles of repose between 30-34 degrees.

Solution processes usually occur when quartz and water are in contact. The entry of the bitumen into the system would probably be to slow down or curtail these processes. Such an inhibition effect was noted by Philipp et al, (1963), in the oil rich sandstones of northwest Germany. The effects of diagenetic alteration of the sand grains were found to decrease with increasing bitumen content.

(e), clay cementation; in the McMurray Sandstone, kaolinite appears to be the predominant clay mineral. However, some illite is also present. Geotechnical problems are greatly reduced since the occurrence of montmorillonite is rare.

The clay minerals kaolinite and illite, since present only in small quantities, would not be expected to contribute appreciably to strength through clay cementation. However, some interesting problems do arise regarding kaolinite.

In situ recovery methods of bitumen employ extraction through a hot water process. Temperatures of between 250-300°C are used. Reaction of kaolinite with other minerals during this process involves the production of montmorillonite, for example,

(a), Dolomite + Kaolinite + Quartz = Calcite + Montmorillonite + CO_2 .

and (b), Calcite + Kaolinite + Quartz = Montmorillonite + H_2O + CO_2 .

Production of large quantities of montmorillonite may be adverse to the hot water extraction process, (Kramers and Carrigy, 1974). From a geotechnical standpoint, presence of montmorillonite involves a host of problems.

However, at higher temperatures, montmorillonite loses its inter-layer water and decreases in volume. Therefore adverse affects of kaolinite converting to montmorillonite may be lessened.

2.4 Test Results From Oil Sand Samples.

2.4.1 Strength Data.

To explain the anomaly between high strength and lack of cohesion, a series of shear box tests were undertaken at the University of Alberta by M. Dusseault.

Twelve circular shear box tests on fine-grained oil sand, where five contained less than 1 percent bitumen and the remaining seven contained from 3.8 to 13.2 percent bitumen, revealed the following:

(a), Extreme curvature of the Mohr failure envelope, Figure (2.5). At low effective normal stresses, 0.49 kg/cm^2 ($1 \text{ kg/cm}^2 = 98.1 \text{ kPa}$), ϕ' values of 56-61 degrees were observed. At higher stresses, 11.4 kg/cm^2 , a ϕ' of 39 degrees was found.

(b), The process of shear invariably showed high dilatancy, the relative amount of which decreased as the effective normal load increases, Figure (2.6).

As seen from the Mohr envelope, a cohesion of zero was obtained eliminating any significant physical cementation as the reason for high strength.

The Mohr envelope for granular soils is not a straight line if plotted accurately. The curvature of the envelope is greatest for dense granular soils and decreases as the soil becomes looser. However, extreme curvature as

shown in Figure (2.5), has never been noted before, to the author's knowledge. From Figure (2.5), the position of maximum curvature of the Mohr envelope occurs at a stress level of approximately 3.0 kg/cm^2 . Comparison with Figure (2.6) also shows that at this stress, maximum curvature of the dilatancy curve results. Hence at this stress level a transition in strength behaviour is recognized.

The correlation of these tests with the natural material is obvious. Along the surface outcrops, where the effective normal stresses are low, slope angles are in the same range as obtained from the Mohr envelope that is, approximately 65 degrees.

The effects of angularity, grading and compaction are well known in the enhancement of peak friction angle. Accounting for these, and noting that the fine-grained oil sands are poorly graded, maximum values of ϕ' lie in the range 35-43 degrees, well below that obtained in the oil sands.

It is apparent that classical soil mechanics concepts are inadequate here and further factors must be taken into consideration.

2.4.2 Thin Sections.

As an aid to understanding the structure of the oil sand, a number of thin sections of the natural material were made, Figures (2.7 and 2.8). These revealed an

interesting fabric: a very high degree of interlock with a significant amount of granular "interpenetration". "Interpenetration" is used here to denote the effect of the concavo-convex contacts, which seemingly show one grain penetrating the other.

In this example of a friable sandstone, the grain aggregating process is entirely one of physical interlock, which holds the grains together as loose pieces of a jig-saw puzzle. The absence of cement is pronounced.

The high degree of interlock is reflected in the bulk density data, which yields figures of in situ bulk density as high as 2.25 gm/cc. It was also found that these densities would not be duplicated under laboratory conditions by any combination of vibratory, tamping or loading techniques (Dusseault and Morgenstern, 1975).

The interlock phenomenon is also shown in the dilatancy results; the process of shear invariably shows high dilatancy, Figure (2.6). As the effective normal load increases the relative dilatancy decreases. This demonstrates that the dominant strength development process at low stresses is one of high dilatancy while at high stress, grain fracture and shear occurs since disruption of the fabric is suppressed.

The mechanism which produces the interlocked structure must be dominated by solution or redepositon of

quartz on the grains. In the former case, removal of mineral matter occurs at the grain contacts. In this particular post-depositional environment, quartz must be unstable and soluble in the permeating solutions. This process is generally known as pressure solution.

In the latter case, addition of quartz to the grains, to produce irregular grain boundaries occurs. The quartz must therefore be supersaturated in the permeating solutions to allow redeposition on the grains. This process is referred to as the production of authigenic quartz overgrowths, or secondary quartz.

Both these processes reflect diagenetic changes in the sediment and are discussed at length in Chapter III.

The photographs of the thin sections of natural oil sand show the inherent interlocking structure as well as individual grain contacts. Point contacts are uncommon. Contacts are either long or concavo-convex but sutured contacts, characteristic of stylolites, are not clearly observable in the thin sections. However, at greater magnification and with better clarity, these contacts may be present.

2.4.3 Electron Microscopy

The scanning electron microscope (S.E.M.) was used to photograph grain contacts in the natural oil sand.

Figures (2.9 and 2.10) indicate that grain

contacts are intimate, with the grains interlocking in a ball and socket fashion. The dilatant behaviour produced during shear at low effective normal stresses is clearly visualized. In thin section, these contacts would be concavo-convex or long.

In these particular examples, the grains are dirty, each covered by a thin layer of clay particles. This is generally not the case in the McMurray as the clay minerals constitute only a few percent in the upper member. Because of this, overgrowths, or their beginnings, are not easily seen. However, Figures (2.11 and 2.12) show the initial stages of quartz overgrowths in the natural oil sand. The figures show the well defined crystal faces associated with quartz growth.

As noted in Chapter III, quartz grains, in an open system environment are usually subjected to varying degrees of solution and pitting. This is mainly due to the chemical nature of the interstitial solution. Figures (2.13 and 2.14) show these features particularly well. These features are not unlike those observed by Krinsley and Donahue (1968) and Pittman (1972).

The above photographs (as well as thin section data), show that the grains in the natural oil sand form an interlocking structure. This is similar to a ball and socket configuration. In traditional soil mechanics concepts, sands are assumed to have tangential contacts (Rowe, 1962).

Therefore resulting shear characteristics are bound to be different. As would be expected in the natural oil sand, dilatancy plays a major role in strength generation at low effective normal stresses.

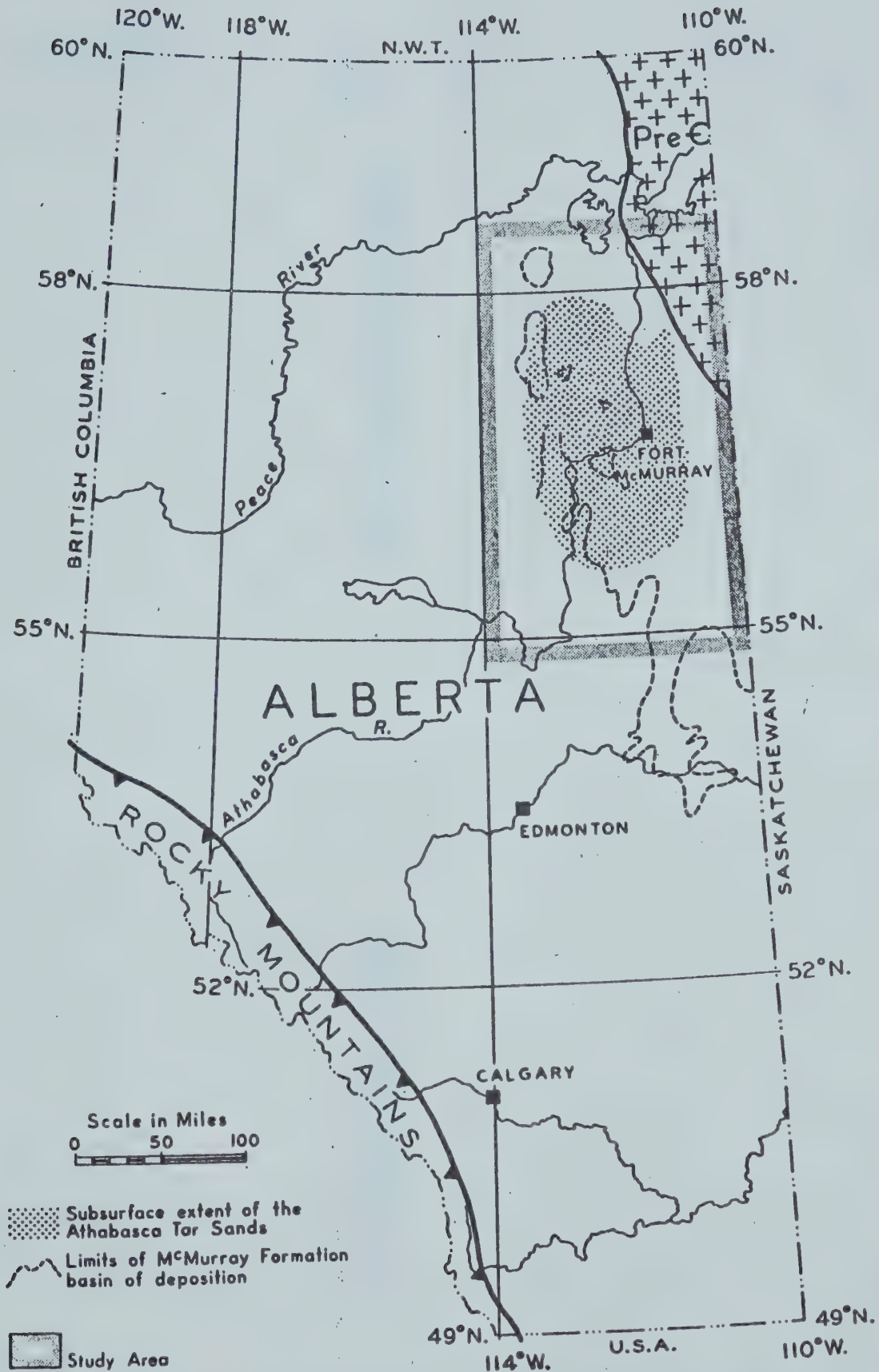


FIGURE 2.1 LOCATION OF THE TAR SANDS
(After Carrigy, 1971)

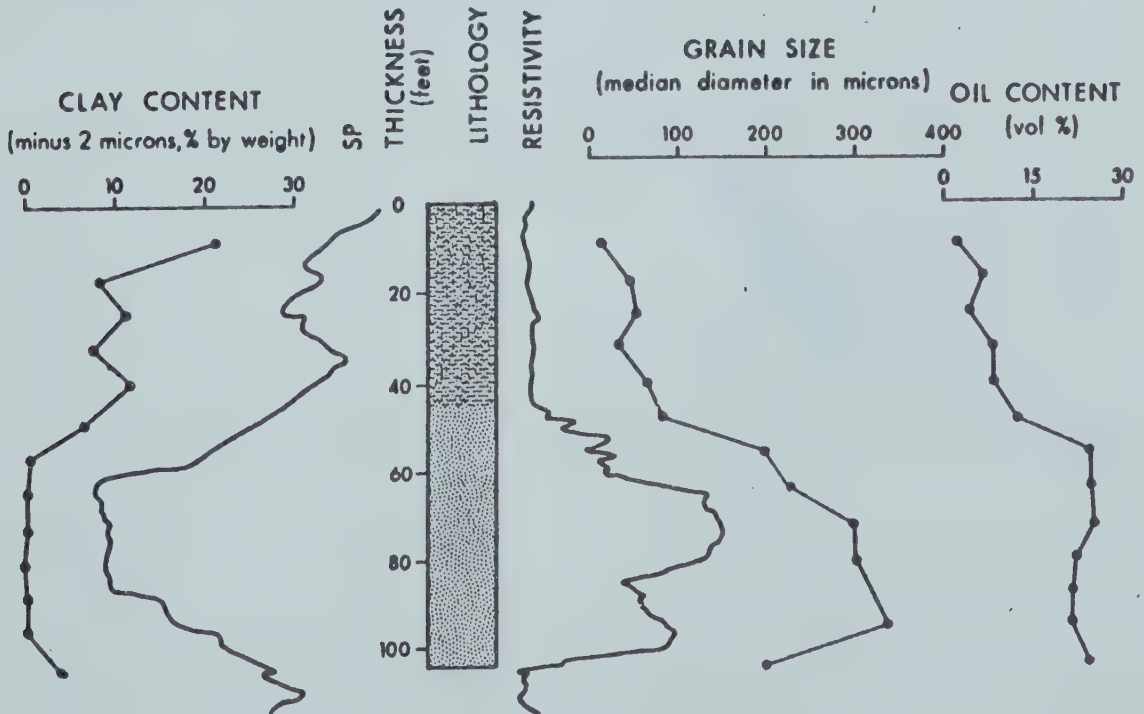
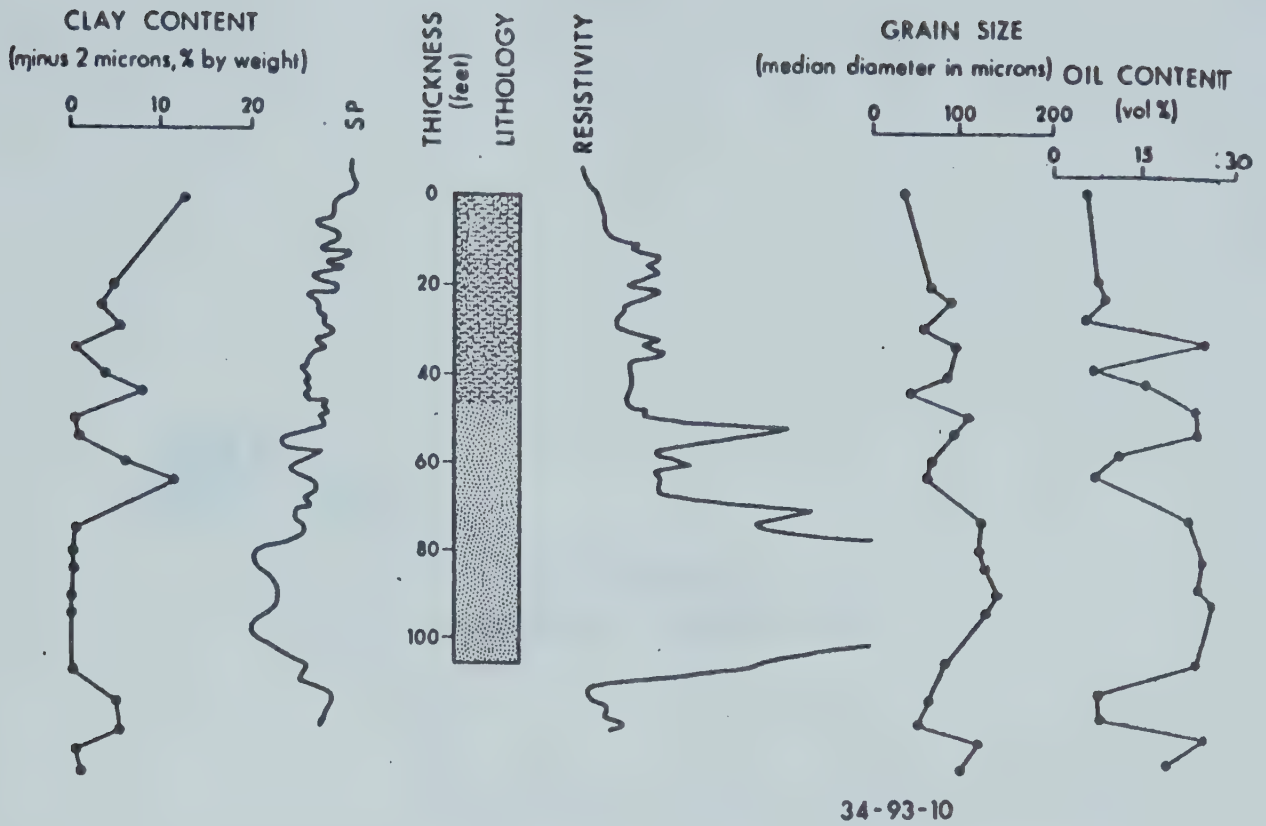


FIGURE 2.2 SIGNIFICANT FEATURES OF THE MCMURRAY
(After Carrigy, 1971)

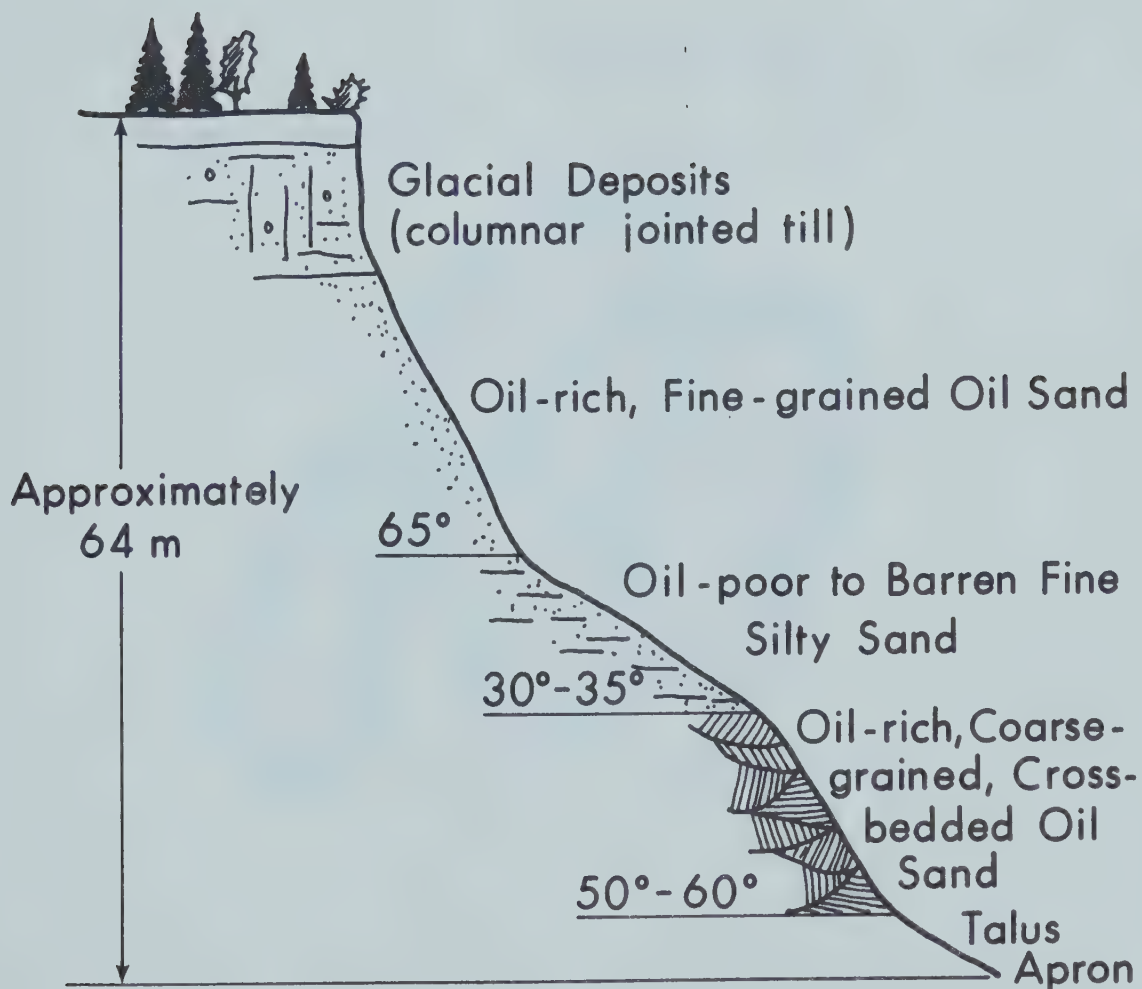


Figure 2.3

TYPICAL OIL-SAND SLOPE

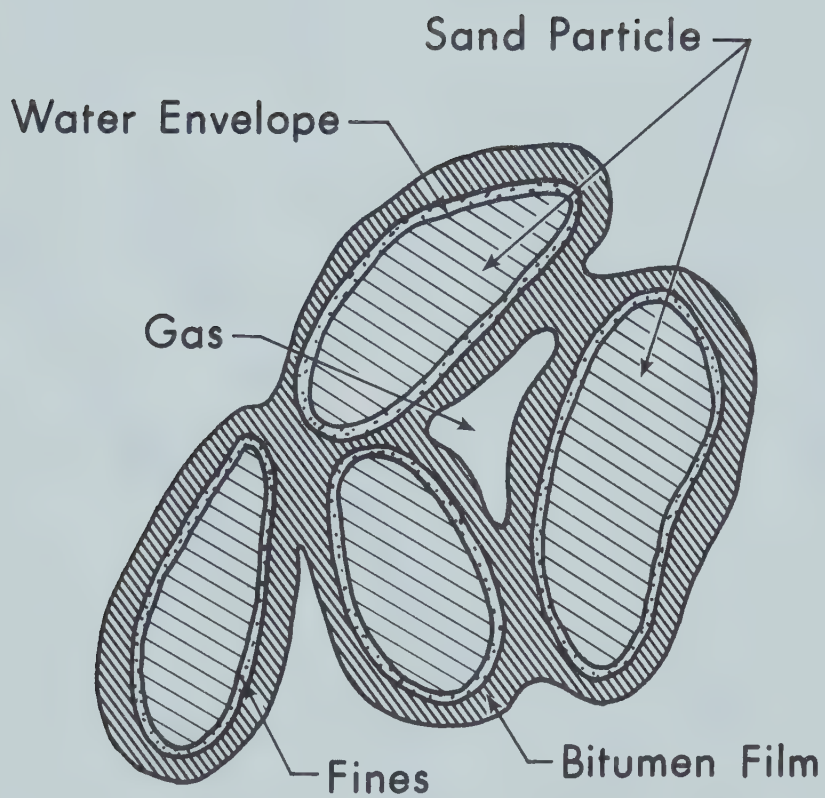


FIGURE 2.4
MAKE-UP OF NATURAL OIL-SAND

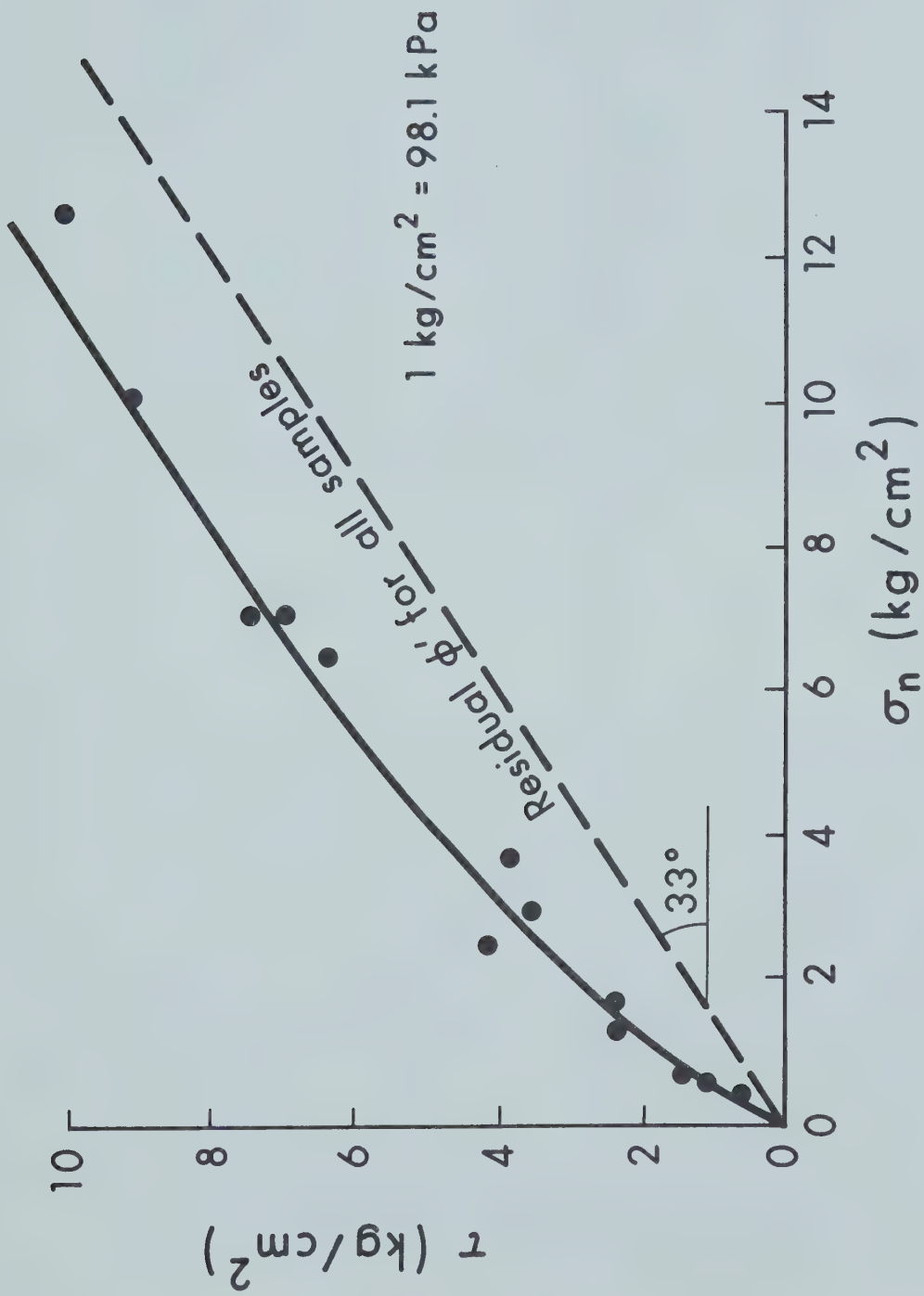


FIGURE 2.5

SHEAR CHARACTERISTICS OF THE NATURAL OIL-SAND

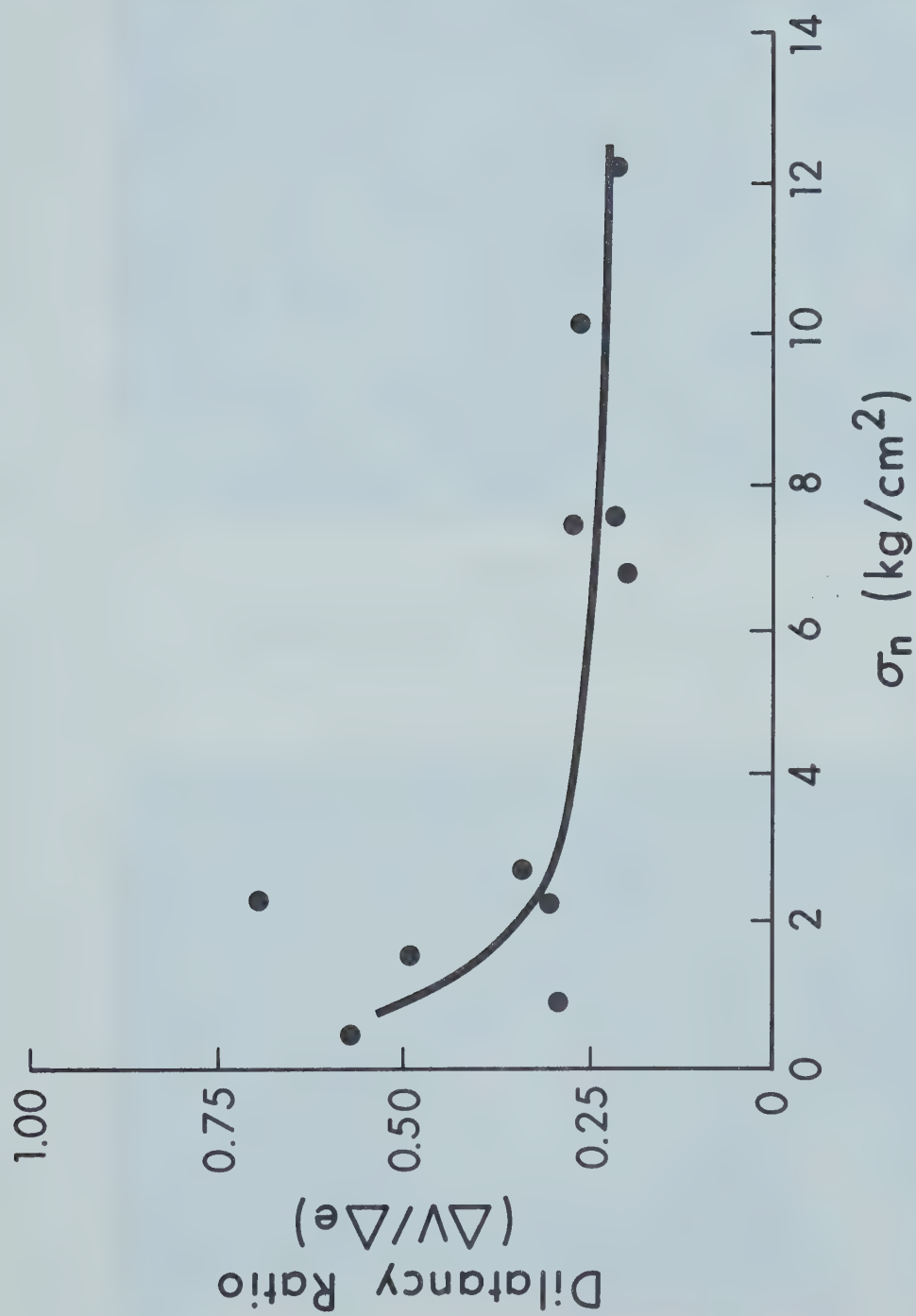


FIGURE 2.6

DILATANCY RESULTS OF THE NATURAL OIL-SAND

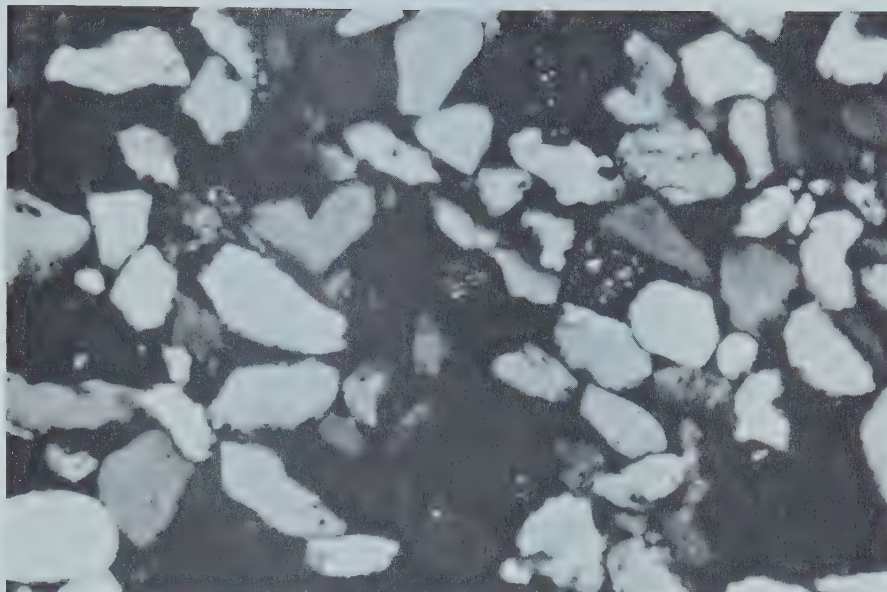


FIGURE 2.7

THIN SECTION OF NATURAL OIL-SAND

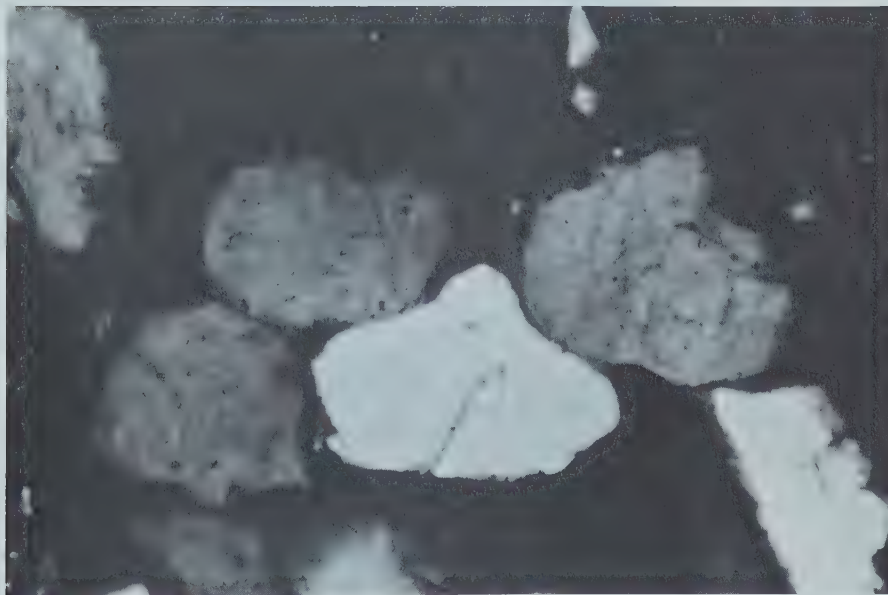


FIGURE 2.8

THIN SECTION OF NATURAL OIL-SAND

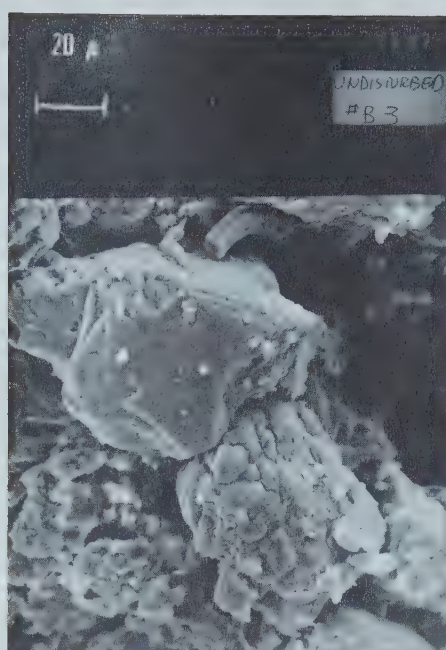


FIGURE 2.9

S.E.M. OF THE NATURAL OIL-SAND

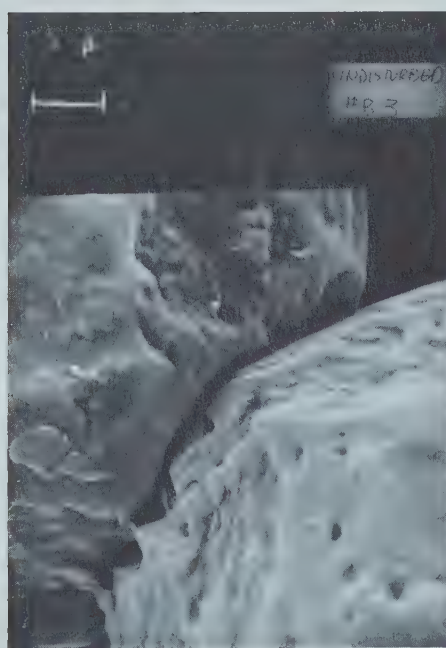


FIGURE 2.10

S.E.M. OF THE NATURAL OIL-SAND

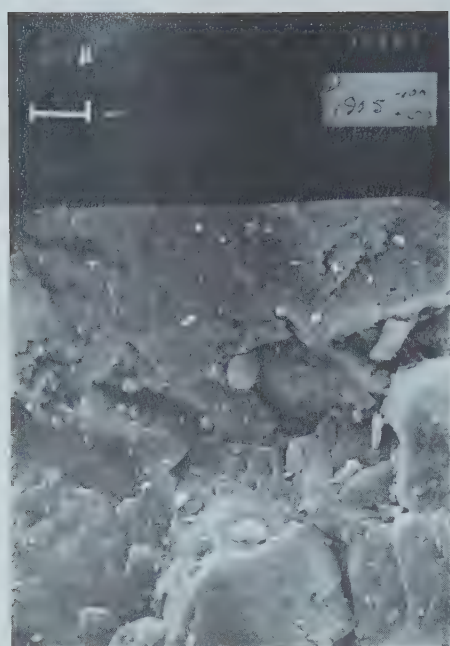


FIGURE 2.11

QUARTZ OVERGROWTHS IN NATURAL OIL-SAND

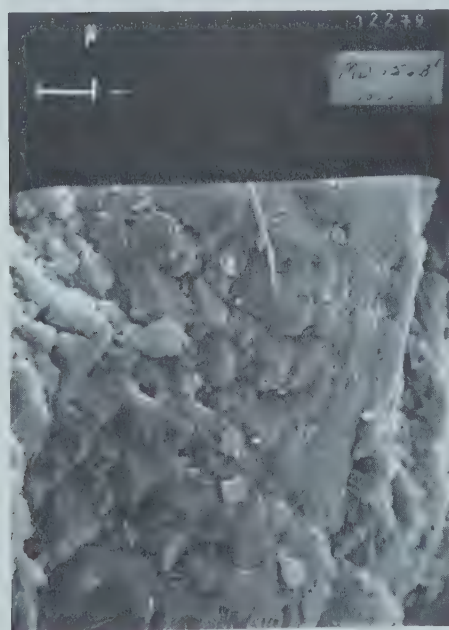


FIGURE 2.12

QUARTZ OVERGROWTHS IN NATURAL OIL-SAND

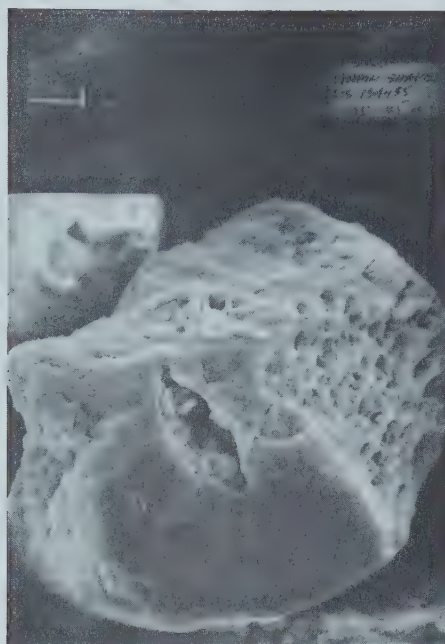


FIGURE 2.13

SOLUTION/PITTING FEATURES OF NATURAL OIL-SAND

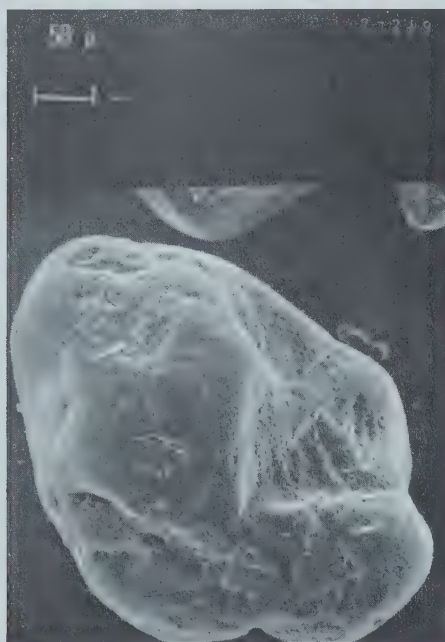


FIGURE 2.14

SOLUTION/PITTING FEATURES OF NATURAL OIL-SAND

CHAPTER III

DIAGENETIC PROCESSES

3.1 Introduction

This chapter deals with the diagenetic processes effective in reducing the porosity of many sandstones. First however, terminology regarding these processes is clarified. Especially confusing is the ambiguity associated with diagenesis and metamorphism. As an aid to understanding the diagenetic processes which occur in sandstones, the geochemistry of silica is examined.

The next two sections deal with these diagenetic processes, namely pressure solution and the development of authigenic overgrowths. Factors affecting these processes and mechanisms involved in their production are thoroughly discussed. Finally, the influence of these processes in reducing the porosity of natural sandstones is examined. Particular reference is made to the McMurray and St. Peter Sandstones.

3.2 Diagenetic Processes

3.2.1 Terminology

The term "diagenesis" is commonly used in the geological literature. However, in the geotechnical literature, it is seldom used. Many varied definitions may be assigned to it (Pettijohn et al., 1973). In this thesis, we restrict its usage by the following definition:

"diagenesis is to include all those chemical processes which effect the sediment after deposition and up to the lowest grade of metamorphism."

In this context, we consider sediment as being made up of detrital minerals, specifically quartz. Examples of diagenesis would include dissolution, redeposition and cementation of the sediment. There is no simple and nonarbitrary dividing line between diagenesis and metamorphism in the pressure-temperature field. It is not unusual to find sandstones in which there have been diagenetic changes related to moderate pressure and temperature increases transitional to rocks in which the pressure and temperature changes were sufficiently high that the geologist conventionally calls them metamorphic.

In practice, there is no great difficulty in distinguishing between metamorphic and sedimentary rocks where the degree of metamorphism is high, or the degree of

diagenesis is low. On the other hand, there are low-grade metamorphic rocks or high-grade diagenetic rocks in which there is no clear cut separation. Therefore, metamorphism and diagenesis should be identified by the types of processes prevailing during these two stages of transformation of primary sediments. This way it will be possible in almost every practical case to distinguish between diagenetic and metamorphic changes.

In the diagenetic zone, the sediments contain interstitial fluids as continuous phases which can be moved by normal flow. These fluids, (most commonly aqueous solutions) are therefore, always involved in the diagenetic reactions, the transport of substances being effected through flow or normal diffusion. Because of the intercommunication and mobility of the interstitial fluids, open-system reactions prevail.

The diagenesis stops at a depth where in all sedimentary rocks the intercommunicating pore spaces have been closed up by physical (for example, compaction) or chemical processes. In this zone, metamorphism begins, its reactions taking place in the solid state or by diffusion at grain boundaries. There are metamorphic reactions of the open-system type, for example, metasomatism. On the whole however, in metamorphism, reactions are closed-system and equilibria between solid phases prevail.

Solutions are to be found in the metamorphic zone

also. They consist of remains of interstitial solutions augmented by solutions released during metamorphic transformations. They invariably collect in places where, through tectonic movements, fractures and cavities have opened.

During diagenesis, the intercommunicating porosity enables the interstitial solutions to penetrate and flow through the sedimentary rock, and thus participate in all diagenetic processes. A knowledge of the chemistry of these interstitial solutions or formation waters is very important for the comprehension of diagenesis. Unfortunately, not enough information is available regarding the principal constituents in these waters. Also, since some of the sandstones concerning us are Ordovician in age, knowledge of water constituents may be purely guesswork. However, before we look at some of these diagenetic processes in detail, some insight into the geochemistry of silica is necessary.

3.2.2_Geochemistry_of_Silica

Silica (SiO_2) is by far the most abundant oxide in the earth's crust. It accounts for over 44 percent by weight of the average sediment (Mason, 1966). In some sandstones, this value may be in excess of 99 percent. It is also one of the most stable minerals.

When dealing with the geochemistry of silica, a distinction has to be made between the crystalline and amorphous forms.

The only crystalline modification of SiO_2 that can form in low temperature and pressure environments is α -quartz. Both α -quartz and the high temperature β -quartz are the most common individual minerals in sediments. Tridymite, cristobalite or coesite represent crystalline modifications of SiO_2 other than α - and β -quartz, but are only of interest in high temperature and pressure petrology. They have practically no significance in the area of sedimentology. Crystalline silica often appears as authigenic microcrystalline quartz known as chalcedony. Subvarieties of chalcedony include agate, jasper and carnelian. Chert, one of the most common siliceous rocks, is principally composed of microcrystalline quartz; small to moderate amounts of amorphous silica however, may co-exist with the quartz. The amorphous and crystalline modifications are mixed in such a way which suggests that the latter may have been derived from the amorphous substrate. Siliceous materials composed of amorphous silica are relatively pure in SiO_2 . They are of extremely small size and arranged in a crystallographic disordered state. This is evidenced by the lack of a characteristic X-ray diffraction pattern. One of the more commonly adopted names for this variety of silica is opal. Included as amorphous silicas are the different varieties of hydrated and de-hydrated silica gels, silica glass and siliceous sinters. Also, amorphous silica constitutes the skeletal remains of silica-secreting organisms, such as diatoms, radiolaria and siliceous sponges, abundant in

pelagic environments.

Solubility characteristics of these two different forms of silica differ considerably. These are shown as a function of temperature in Figure (3.1), (Siever, 1962). Extrapolation of the temperature-solubility curves to 50°C indicates that quartz would have a solubility of 5.7 parts per million (p.p.m.) and amorphous silica, about 60 p.p.m. Also, the curves appear to converge at higher temperatures and presumably would meet at some temperature close to the critical temperature of water. Kennedy(1950), studied the SiO_2 - H_2O system at much higher temperatures and pressures. Isobaric lines range up to 1750 bars, at temperatures in excess of 400°C. He found the effect of temperature and pressure on silica solubility reinforce each other and make silica much more soluble at higher temperatures and pressures. The effect of temperature is much greater than that of pressure.

Silica will dissolve in strongly alkaline solutions. Figure (3.2), (Krauskopf, 1959), shows that solubility is essentially independent of pH for values below 9.0, but rises abruptly at higher values. Quartz is insoluble in all acids except hydrofluoric (Berry and Mason, 1959). It has been shown by Okamoto et al. (1957), that the pH at which the solubility of silica increases abruptly is independent of temperature up to 100°C.

In natural waters (pH below 9.0), silica may be of

two possible forms, (after Degens, 1965) ;

(i), molecularly dispersed - that is,
as a true solution,

and (ii), in a polymerized or colloidal state.

A silica solution must be supersaturated with respect to amorphous silica before precipitation of a gel or colloidal particles (that is, amorphous precipitate) can take place. Theoretically, it is possible to expect quartz to crystallize from a solution supersaturated with regard to its solubility product. This however, is not the case, as evidenced by the lack of syngenetic quartz precipitates in recent and ancient environments. In nature, this is due to the extreme slowness of the reaction. A long time is also necessary for the conversion of the amorphous silica precipitate to quartz. Moreover, the progressive nature of the change as observed is ;

opal \longrightarrow chalcedony \longrightarrow quartz.

High temperatures, in excess of 150°C, are required to produce quartz in the laboratory, within a reasonable time. The outstanding characteristic of almost all natural waters is their apparent undersaturation with respect to silica, for example :

(a) Rivers and Lakes ; the majority of rivers and streams fall into the range from a few parts per million of dissolved silica to 35 p.p.m. A very small number range from 35-75 p.p.m. It is generally found that the silica content will be highest in the head-waters of streams that drain

from a metamorphic and/or igneous terrain. As streams travel onto their middle and lower courses, they become more diluted by run-off waters low in silica. Consequently, the silica content is diminished.

Lakes may have relatively high concentrations of dissolved silica. This is particularly true of alkaline lakes in which, due to high pH, solubility of silica may be much higher (Clarke, 1924).

(b) Groundwaters ; it is usually found in groundwater analysis that in the upper zones of fresh water there is little silica in solution and the solutions are vastly undersaturated. (Meents et al., 1952). However, in deeper groundwaters, concentrations may rise much higher. Added to this is the rise in temperature due to the geothermal gradient, and in such cases, saturation of silica may occur. If there is some change in conditions, say a temperature decrease, precipitation of amorphous silica would result.

(c) Oceans ; oceans are undersaturated with respect to amorphous silica. Silica concentrations are very low, usually less than 1 p.p.m. with a maximum of 4 p.p.m. Since the great bulk of the oceans lie within a temperature range from 2-5°C, and referring to Figure (3.1), open sea waters are only slightly undersaturated with respect to quartz, although something like 1/10 saturated with respect to amorphous silica. Biochemical action is responsible for keeping the oceans so low in dissolved silica, for it is apparent that quartz precipitation is too slow near 0°C.

Diatoms, radiolaria, siliceous sponges and other silica-secreting organisms are efficient in extracting silica from solutions undersaturated with respect to amorphous silica. The diatoms can precipitate silica from solutions even at very low concentrations, less than 1 p.p.m. (Siever, 1962).

Having dealt with characteristics of natural waters in terms of concentrations of dissolved silica, the sources of silica in solution are now examined:

(a) Weathering of Silicates ; this is the primary source of silica in solution. Chemical degradation of these minerals by water solutions results in varying amounts of SiO_2 in solution. Amounts are dependent upon climate, relief and the nature of the original material.

The silicate minerals of igneous and metamorphic origin, feldspars, amphiboles and pyroxenes, that break down rather rapidly under soil forming conditions may be expected to supply the greatest proportion of silica in solution (Siever, 1959). Quartz from igneous and metamorphic rocks and from quartzose sediments, considering its low solubility and slow rate of solution, probably supplies a less significant amount.

(b) Weathering of Clays ; the weathering of clay minerals provides another source for silica in solution. It may be shown (Siever, 1962), that the transformation of montmorillonite into muscovite, kaolinite and/or illite, results in substantial quantities of silica in solution.

Clay mineral-silica reactions are affected by pH. Lowered alkalinity would displace the montmorillonite-kaolinite reaction toward the production of kaolinite and silica.

(c) Thermal Spring Waters ; another source of silica in solution is from thermal spring waters. Generally, because of the higher temperature associated with such waters, they are greatly enriched with silica. As the waters cool off, they may release silica in the amorphous form, for example, siliceous or opaline sinters.

Solubility of silica is greatly increased by rise in pressure and/or temperature. Deep formation waters therefore may be expected to be supersaturated with respect to silica. Migration upwards of such with resulting cooling would cause precipitation of silica.

(d) Biochemical Sources ; dissolution of siliceous organisms in early diagenesis of sediments provides a major source of silica. These organisms can extract huge quantities of silica from sea water and, during life, protect their siliceous parts from dissolving in the sea water. Some time after death, and cessation of metabolic processes that protect the silica organisms from dissolution, the organisms slowly dissolve in the undersaturated solutions around them.

High concentrations of dissolved silica exist in the interstitial waters of the bottom sediments of the Bering Sea (Siever, 1959). Also, further examination have revealed corroded and partially dissolved diatom shells in

the sediments.

(e) Water of Compaction ; reverse osmosis is used to explain the increase in concentration of salts in a brine solution beneath a semi-permeable membrane, Figure (3.3), (Siever, 1962). As the salt solution passes through, the fluid becomes more concentrated beneath the membrane and less concentrated above the membrane. If the fluid were one containing dissolved silica, and the semi-permeable membrane taken as a shale bed, then a greater concentration of silica would build up below the shale bed, say in a sandstone bed. Supersaturation would occur and precipitation results. Above the shale bed, the solutions are undersaturated and only solution can take place.

Such a mechanism has been noted in the laboratory (McKelvey and Milne, 1960), and implied by determinations of salinities in modern sediment interstitial waters (Siever et al., 1961). It is thought reasonable that such a mechanism may occur if the fluid contains dissolved silica, rather than dissolved salts.

3.2.3 Pressure Solution

Pressure solution is important in the compaction of detrital sediments. It is an effective agent of bulk volume reduction through the gross dissolution of the detritus. Dissolution takes place at grain contacts. Pressure solution is a diagenetic process.

The basic idea of pressure solution with respect

to quartz, is that high effective pressures are developed at point contacts of grains which therefore become more soluble. These point contacts dissolve preferentially. The dissolution liberates dissolved SiO_2 to the pore water. If the pore waters are undersaturated with respect to silica, and free movement of these waters can occur, then the silica is taken out of the sediment. On the other hand if the waters are supersaturated and movement is confined, the redeposition of silica will occur. This is essentially the mechanism in the production of authigenic overgrowths.

Pressure solution is characterized by the interpenetration of one grain into another. The resulting structure is a prime cause of porosity reduction. The nature of the grain contacts, ranging in degrees of interpenetration from tangential through concavo-convex to sutured, Figure (3.4), has been related to porosity reduction and depth of burial, (Taylor, 1950). A net increase in the number of contacts per grain with depth was found, for example, 2.5 contacts per grain at 3000 feet, compared to 5.2 contacts per grain at 8500 feet. An increase from tangential to concavo-convex to sutured contacts with depth was also found. However, in Taylor's study the sandstones had a quartz content ranging from 25-65 percent and were rich in lithic fragments and other ductile grains. The systematic increase in grain contacts was a result of deformation of soft fragments, as well as solution of quartz grains. Thus Taylor's study is not entirely applicable to

pressure solution versus depth problem, but merely indicates the general trend.

Further evidence of pore space reduction that is related to pressure solution is the measurement of what Heald (1956) called the "minus-cement porosity", the porosity which would be present if a specimen contained no chemical cement. Minus-cement porosity measurements on many sandstones indicate significant compaction that would not be accomplished by simple mechanical arrangement of the grains. It therefore must be due to solution.

Pressure solution features are evident to some degree in most sandstones. Attempts to classify the geometry of these features have been made by Trurnit (1968). Stylolites are particular examples of pressure solution features. A stylolite is a complex interface between two rock units which exhibits mutual column and socket interdigitation. The long axes of the columns and sockets are perpendicular to the interface and are generally parallel to one another. The interspace between the rock units separated by a stylolite commonly is characterized by a thin seam of insoluble residue which became concentrated as the rock was dissolved.

Stylolites may range from microstylolitic grain contacts, a few tenths of a millimetre in amplitude, to one or two metres in amplitude, (Park and Schot, 1968). They may be traced over distances extending 50 metres or more.

Stylolites are found in some sandstones and carbonate rocks, but are most common in limestones.

Some factors which effect pressure solution include :

(a) Structural Deformation ; Siever (1959), investigated Pennsylvanian Sandstones from two areas believed to have been buried to the same depth, but with different degrees of structural deformation. Quartz grains in the intensely folded sandstones were highly presolved compared to quartz grains in the slightly deformed sandstones.

In comparison of samples buried to different depths with the same degree of structural deformation, he concludes that the effect of structural deformation was more pronounced than the weight of overburden.

An explanation for this may be due to the fact that during structural deformation, temperatures may be increased due to shear processes and downwarping of the strata. Solubility would therefore be increased too. It is interesting to recall from the experiments of Kennedy, (1950), that temperature had a greater effect on quartz solubility than pressure.

(b) Grain Size ; as theorised by Weyl (1959), reduction in volume from pressure solution varies inversely with respect to the cube of the grain radius. Heald (1956), illustrates in the St. Peter Sandstone that porosity in a fine-grained bed has nearly been eliminated by pressure solution while a

few centimetres away in a coarse-grained bed, considerable pore space remains.

A random mixing of grain sizes (poor sorting) will lead to preferential pressure solution of the finer grains, accompanied by a readjustment of the positions of the coarse grains. Also important is the grain size variation in the direction of the maximum principal stress (Weyl, 1959).

Renton et al., (1969) showed through experimental work that fine-grained sands are more susceptible than coarse-grained sands to destruction of porosity by pressure solution and growth of secondary quartz.

(c) Clay, iron oxide or carbonaceous coatings ; grains with laminae or coats of clay, carbonaceous material, mica or iron oxide, seem to greatly facilitate pressure solution (Weyl, 1959, Fuchtbauer, 1967).

In the case of clay coatings, one explanation involves the release of potassium ions by illitic clays and resultant formation of an alkaline microenvironment between the grains and solution (Thomson, 1959). Weyl (1959), proposed that pressure solution was favoured by greater diffusion through the clay particles among the grains. Heald (1956), speculated that the clay acted as a catalyst.

Lerbekmo and Platt (1962), outlined a similar alkaline microenvironment situation as Thomson (1959). However, in this case, the reduction of iron sulphide with

the release of OH^- -ions caused the increase in alkalinity.

It is interesting to note that Siever (1959), in his study of the Pennsylvanian sandstones did not find any positive correlation between clay and pressure solution. However, he was dealing with a more argillaceous (up to 30 percent clay) sandstone, rather than clay coatings on grains.

A correlation may be drawn with stylolites on this point. Stylolites with thick clay coatings are very rare while stylolites with thin clay coatings are common (Fuchtbauer, 1972). A reason for this may be due to the fact that a thick clay coating of clay will diffuse any solution passing through it, Figure (3.5). On the other hand, a thin coating of clay will tend to concentrate the flow along a particular narrow path. Thus stylolites having only a thin coating of clay will be more easily formed.

(d) Oil, gas and other organic impurities ; it is commonplace to find oil and gas deposits in sandstones. As shown by Philipp et al., (1963), the presence of oil will inhibit quartz diagenesis. Fuchtbauer (1974) shows that the percentage of quartz overgrowths in a water-filled sandstone compared to that in an oil-saturated sandstone, is considerably higher. Only adjacent to the shale did the number of quartz overgrowths in the oil-saturated sandstone increase markedly.

siever (1962), showed that the solution of

amorphous silica in peat waters was extremely small (approximately 1 p.p.m.) whereas in distilled water, solubility is 140 p.p.m. The simplest explanation for the anomalously low results is that colloidal organic compounds in the peat water were absorbed on the free silica gel surface and prevented that surface from fully saturating the solution.

(e) Temperature ; this is an important variable when dealing with the dissolution of quartz. To the Author's knowledge there is no data relating temperature to quartz diagenesis. Although temperature data pertaining to deep boreholes are known (Dapples, 1959), it may not be the temperature at which pressure solution features formed.

(f) Age ; this is another difficult factor to evaluate. The age of the rock may have no relationship with the actual time the rock was exposed to the physico-chemical conditions necessary to produce pressure solution.

Mizutanti (1970), determined, by natural observation and experiment, the effects of both temperature and age (to 1 billion years) on the effects of quartz in Japanese rocks. For deep sea sediments, it can be concluded from his data that age and not temperature plays the most important role in silica diagenesis. Beall and Fisher (1969) studied Miocene (25 m.y.) deep sea clastics from a 100 metre maximum depth of burial. Their sediment composition, in terms of silica diagenesis, was in close accord with Mizutanti's, thereby supporting its validity.

For the process of pressure solution to continue, stress must be passed across the contact from grain to grain, while a solution film is simultaneously maintained between the grains so that dissolved ions may diffuse through it to sites of lower stress. Two mechanisms involving this concept have been proposed :

(a) The undercutting mechanism (Bathurst, 1958) calls for the establishment of an irregular surface between the grains at a microlevel. Solution is visualized as taken place over only a small part of the surface at one time, undercutting higher relief portions of the surface, Figure (3.6).

and (b) The solution film mechanism (Weyl, 1959) calls for the maintenance of a continuous film of solution between the grains which is able to support shear stress and allow diffusion of the ions, Figure (3.7). The maintenance of the solution, it is argued, is implied in the force of crystallization, noted in experiments. This is the term given to the force exerted by a growing crystal when constrained.

Other mechanisms have been proposed, these include;

(c) The possibility of microenvironments of high pH at the contact of quartz grains (Thomson, 1959). Reduction of potassium in illitic clay surrounding some of the grains may result in the formation of potassium carbonate, a strong alkali. As the solubility of quartz increases with pH over 9.0, under conditions of uniform stress, dissolution will

begin first in the zones of high pH. If a pH gradient of decreasing magnitude radiates from these zones of clay, then redeposition may occur in zones of low pH.

and (d) Lerbekmo and Platt (1962) suggest that the presence of carbon and iron compound coatings promote pressure solution. This is caused by a reduction of the iron to produce iron sulphide and/or iron carbonate, with the subsequent release of OH^- -ions. This will increase the pH of the solution at these contact points (as in (c)) and thus increase the tendency for quartz to dissolve.

Any of the mechanisms, (a)-(d), are difficult to rule out. The first mechanism suffers from the disadvantage that it does not explain the phenomenon of "force of crystallization", (Becker and Day, 1916, Taber, 1916), which is essentially the antithesis of pressure solution. One does not see evidence of it in these sections; however, one might argue that the undercut area is too small to be seen.

Mechanisms (c) and (d) are local effects. However, neither one explains why pressure solution occurs when clay or carbon/iron compounds are absent. According to Okamoto et al. (1957), the solubility of amorphous silica is increased at 71°C beginning from pH 9.0, at 200°C beginning from pH 8.0. LeRibault (1971), showed that many quartz grains in sandstones have an amorphous SiO_2 -coating of about 3 μm thickness, which may explain at least part of the pressure solution. The necessary parameters of this reaction are

elevated temperature, low water mobility, (to prevent dissipation of the exsolved K-ions or OH^- -ions), and time. Pressure however, would not be a critical parameter.

In mechanism (b), the most critical assumption involves the mechanical properties of the solvent film, that is, the change in thickness of the film with effective normal stress. Weyl examines two cases : (i), a film thickness independent of the effective normal stress and (ii), an exponentially decreasing stress/thickness relationship.

His results indicate that in the first case the rate of compaction is directly proportional to the average effective stress and inversely proportional to the square of the radius of the contact area. The second case shows that the rate of compaction will increase at a rate slower than linear with the average stress. Needless to say the behavior of such thin films is not well understood. Other important factors, as well as thickness are the rate of diffusion in and out of the film and the temperature.

Weyl does not adequately explain the observation that stronger pressure solution is observed between quartz/clay/quartz contacts than at clear quartz/quartz contacts. The opposite should be expected since according to the "Riecke Principle", stressed lattice areas are more soluble than unstressed areas.

Pore volume is reduced by pressure solution as a result of closer packing of grains, grain corrosion and filling of the pores with cement generated by pressure solution. Pittenhouse (1971,a), demonstrates the degree of pore space reduction to be expected by solution of grains at points of contact for several ordered packing arrangements of spheres. The volume of cement which could be generated by pressure solution was also estimated. Since the original porosities for ordered packing arrangements of spheres are known, and pore volume is calculated on the basis of bulk volume minus grain volume, then the degree of pore volume reduction can be expressed as a function of decrease in bulk volume, Figure (3.8). The data for this model were obtained following the method devised by Rittenhouse and do not reflect the added influence of void filling cement. Figure (3.8) shows the degree of pore volume reduction (as a percent of original porosity lost), plotted against the bulk volume reduction owing to pressure solution for cubic and orthorhombic regular packs of spheres, curves A and B (respectively). A reduction of 40 percent in bulk volume causes a 35 to 44 percent reduction in pore space without cementation, depending on the packing arrangement.

It has also been observed (Dunnington, 1967), that noticeable decrease in porosity has been found in the immediate vicinity of stylolites.

As well as a process involving gross dissolution,

pressure solution may play a significant role in providing a source of cement, commonly known as second generation cement. For a high degree of silica saturation in pore solutions, it is not likely that pressure solution generated cement could travel far before being precipitated in the available pore space. Accordingly, the amount of dissolved silica which is being produced must exceed the remaining pore space volume in order for the cement to be exported from the immediate vicinity.

Coogan and Manus (1975), show the relationship between bulk volume reduction and the ratio of cement generated to the remaining pore volume. A comparison was made for regular cubic and orthorhombic packs of spheres according to the method used by Rittenhouse (1971,a). In addition, data were generated for a regular pack of cylinders arrayed so that the ends of the cylinders appeared stacked in a cubic regular order. As seen from Figure (3.9), the available pore space exceeds the volume of produced cement until a point is reached where the cement generated (CG), divided by the pore volume remaining, equals unity. There the bulk volume is reduced by an amount equal to the original porosity. Indeed, in the case of cubic and orthorhombic packs of spheres, curves A and B (respectively) in Figure (3.9), the bulk volume reduction would have to be in excess of that commonly supposed to occur before allochthonous cement could be generated. Of course, this assumes local precipitation of cement so long as pore space

is locally available. In the case of cubic packs of cylinders, curve C in Figure (3.9), the original porosity (21.5 percent) is lower and the bulk volume loss required is more in keeping with most estimates of volume reduction.

In general, Figure (3.9) sheds doubt upon the efficiency of the general process of pressure solution - generated cement. However, in the expulsion of water from muds into sands, this process may be important.

The association of augmented silica cement adjacent to clay beds is a common one. Fuchtbauer (1967), showed how quartz cementation in a Dogger Sandstone increases towards the shaly margin of the bed and ascribes it to an infiltration of water from the shale into the sandstone. Because the largest part of water of compaction leaves muddy sediments relatively early in their diagenetic history, it is likely that the source of silica supersaturated water is important in the early stages of sandstone diagenesis (locomorphic stage), before deep burial has taken place. Fuchtbauer (1972) later recognizes that there will not be enough cement produced to account for the tremendous volume of quartz overgrowths present in the ancient rock record. Consequently, to account for silica cementation and pressure solution, one must consider local chemical gradients and resultant large scale, short distance material transfer.

Since it is considered that the ions derived from

pressure solution travel in fluids parallel to the direction of maximum stress, it is tempting to suggest that pressure solution is a self-limiting process.

Much work has been done using electron microscopy to examine the surface features of sand grains, (Krinsley and Donahue, 1968, Margolis, 1968, Krinsley and Margolis, 1968, Pittman, 1972, and Ingersoll, 1974).

Two main categories of diagenetic surface features have been observed and are commonly associated. The first of these is a wavy, etch pattern ; the second is a worn low relief solution surface. As older and older sediments are examined, diagenesis tends to become more pronounced and mechanical textures are eliminated.

The etch pits on quartz have been described by Amelinchx (1964) as being an expression of dislocations and defects within the crystal. The size and number of the etch pits has been found to be a function of the relation of the dislocation or defect within the crystal to the surface. When a spiral dislocation intersects the crystal surface, the dislocation will be expressed, after etching, as an oriented pit. Solution is initiated at the site of the dislocation or defect since the quartz is more soluble there. The increased reactivity is caused by the strain that is present.

Coalescence of etch pits in time would produce a worn, low relief solution surface.

Pittman (1972), examined the points of contact between quartz grains. He found them to be sites of pressure solution. They appeared as circular to ellipsoidal spots marking points of contact between visible grains and formerly adjacent grains which had been removed along a fracture surface during sampling. The presolved surface varied from areas that were relatively smooth to areas that consisted of ridges and knobs with corresponding furrows and pits. Some presolved surfaces have a radial or linear pattern of ridges and furrows. It is suggested that these furrows serve to channel solution into and out of the presolved area. Renton et al. (1969), produced similar ridge-furrow features experimentally.

The central portions of a moderately presolved area commonly consist of irregular ridges and knobs with corresponding furrows and depressions. The elevated area of one grain fits into the corresponding depression of the adjacent grain or partner involved in the pressure solution process. Pittman found it difficult to obtain a highly presolved sutured surface for observation because the rock fractures across grains rather than around them.

As stated before, interpenetration occurs at the site of a defect on the surface, that is, where the quartz is more soluble. It is also found when two spherical grains

of identical composition pit each other through pressure solution, the smaller grain always tends to protrude into the larger one (Park and Schot, 1968). This is apparently attributed to differential surface energy effects.

Also, if the hardness of quartz was anisotropic, interpenetration would more readily occur under certain favourable conditions.

3.2.4 Authigenic Overgrowths

As noted before, silica in solution may originate from a variety of sources. Consequently, the precipitation of silica from solution is not an unreasonable process. This may result from a change in chemical conditions. Precipitation of silica is therefore a diagenetic process.

One of the most common precipitates is the addition of quartz to detrital grains. (Whether this is the amorphous form of silica or not is irrelevant, since it will revert to quartz with the passage of time). As the amount of quartz added to the grains is increased, lithification of the aggregate is achieved. Continued precipitation results in a quartzite texture primarily by welding of one grain to another as the line boundaries are developed. This is quite distinct from the texture produced in which the grains touch one another with tangential or point contacts, and are held in position by simple cement precipitated in the interstitial pore space. In the former case, the resulting texture shows slight interlocking of grains and the

aggregate ranges in degree of friability in inverse proportion to the amount of precipitated overgrowth.

Since the amount of the precipitated material is small even in concentrated solutions, continued precipitation could not have come from a closed system without replenishment of solution. Rather, it must have come from a steady supply of supersaturated solution flowing through the aggregate. As the precipitation process continues, the pore spaces will become smaller, the permeability steadily reduced the flow rates diminished and the rate of precipitation slowed, Figure (3.9), (Pettijohn et al., 1973). Since the initial permeability and its rate of decrease is a function of grain size distribution, a fine-grained aggregate with lower permeability would, in the final stage become lithified before a coarse-grained one. As seen in Figure (3.9), the time to completely lithify an aggregate is very long compared to that needed for partial lithification. Since it is quite difficult to distinguish detrital quartz grains from quartz cement or overgrowths in many sandstones, data relating to geological age is sparse.

Syntaxial overgrowths are those deposited in optical continuity with the original grain. They may be regarded as a resumption of crystal growth albeit under vastly different conditions from that of the original crystal. The mineral that shows this behaviour most commonly is quartz ; feldspar and calcite overgrowths are less common

in sandstones. Minerals precipitated on substrates of different mineralogy form epitaxial overgrowths, such as carbonates on quartz surfaces or clays on quartz.

The precipitation of silica with the resulting formation of overgrowths is not a simple process. Pittman (1972), recognizes two types of incipient overgrowth; (a), growths with poorly defined crystal faces that form an interconnected anastomosing system over the surface of the grain and (b), isolated growths with well defined crystal faces.

In the latter, crystals commonly grow until contact is made with an overgrowth that nucleated elsewhere on the surface of the host grain. The former is characteristic of irregular growths with disorderly growth over the detrital grain.

The size of precipitated crystals is related to rapidity of crystal growth and nature of substrate. In general, small crystals indicate relatively rapid growth and most commonly, large crystals are the result of slow growth. It is frequently noted that an overgrowth starts as many tiny growths on the immediate substrate. As the crystals move away from the substrate they get progressively larger until only a single large crystal grows, Figure (3.11). This can be ascribed to minor fluctuations in individual crystal growth rates whereby the faster growing crystals overtake and encompass the smaller, slower growing ones. For example,

the rate of crystal growth of quartz crystals varies with crystallographic direction and is most rapid in the direction of the C-axis. It may also be a product of the slowing down of crystal growth as the pore space becomes progressively filled, permeability is reduced, flow rate of solution is reduced and crystal growth becomes slower : the consequence is larger crystals.

The presence of "line boundaries" in detrital quartz grains apparently has an effect on the location of overgrowths (Pittman, 1972). The line boundary is a surface of high density dislocations (crystal defects) and consequently, the line along which this surface intersects the surface of the quartz grain is a line of relatively high energy. As such, it is an unfavourable site for new crystal growth. This was observed by Ernst and Blatt (1964), where initial small growths never straddled the line but commonly occurred adjacent to and on both sides of it.

Quartz overgrowths grow into pores in a sandstone until an obstruction is encountered such as an adjacent detrital grain or another overgrowth. An overgrowth molds itself against the surface of an adjacent detrital grain and faithfully reproduces the texture of the rough surface, regardless of the composition.

Studies undertaken by Cecil and Heald (1971), Heald and Larese (1974) indicate that coatings of chlorite, illite, hematite, chert or carbonate may inhibit the

formation of quartz overgrowths. Although these coatings do not always prevent formation of overgrowths, they commonly cause the growths to form with unique features. For example, distinctive hourglass patterns were observed between detrital quartz grains which had thin clay coatings (Heald and Larese, 1974).

The "dust line", (that is , that line separating the detrital grain from the secondary enlargement) is important in the recognition of quartz overgrowths in thin section. The dust line may be some type of coating on the quartz grain, for example, hematite is common. It may be a sequence of intricate voids or a combination of impurities on voids. S.E.M. examination (Pittman, 1972), reveals that the contact between overgrowth and nucleus is open. This means that impurities may, at a later period, get between the overgrowth and nucleus. Sometimes these voids may be filled by liquid. However, secondary quartz usually infills many of these voids which destroys the dust line, unless impurities are also present.

Using current microscopy techniques, distinguishing detrital quartz grains from quartz cement or overgrowths in many sandstones is difficult, if not impossible. Compare Figure (3.12) in which the sandstone is viewed in plane-polarized light (left) and by cathodoluminescence (right). Detrital quartz grains appear to have sutured and concavo-convex boundaries indicating pressure

solution in plane-polarized light. However cathodoluminescence reveals that there is extensive secondary enlargement with little or no pressure solution (Pettijohn et al., 1973).

The voids, present in some dust lines, are not unlike the microcavities observed in some crystalline rocks. Sprunt and Brace (1974), observed that 20 percent of quartz grain boundaries in unstressed Westerly granite were cracked. Special preparation procedures were used to eliminate cracks produced by sectioning. Some speculation to the origin of these cracks was that at one time they were sites of fluid inclusions.

3.3 Correlation with the McMurray and St. Peter Sandstones

Porosity is perhaps the most important property of sandstones. This is especially true in the exploration for new oil and gas reservoirs.

As a general rule, the porosity in coarse-grained sands and sandstones decreases considerably with depth at shallow depths and more slowly at greater depths. In fine-grained sands and sandstones, the opposite is true (Fuchtbauer, 1974). Figure (3.13) shows that at depths of 5000 metres, coarse-grained sandstones will retain a porosity of more than 10 percent. As Fuchtbauer indicates, one explanation for this is the higher roundness of coarse sand grains by which they slide more easily into narrower positions than fine grains, in the shallow realm of mechanical compaction, in as much as the pressure per grain contact is higher in coarse than in fine sands. On the other hand, fine-grained sandstones are subject to dissolution reprecipitation processes at higher depth than coarse sandstones because of the higher specific surface of fine sand and tiny surface protrusions due to poor rounding of such grains.

Traditional soil mechanics concepts on compressibility argue that a linear relationship between change in void ratio and logarithm of effective normal stress prevails. No distinction is made between grain size.

In the realm of chemical compaction below a depth of 1000 metres, Figure (3.13), pressure solution is one of the most important mechanisms in compaction due to (a), reduction of space between grains and (b), by causing the precipitation of silica in solution.

Porosity can also be reduced upon loading by plastic deformation of ductile grains. Rittenhouse (1971,b), showed that the greatest reduction in pore space is achieved when ductile grains are present in an orthorhombic packing configuration.

Temperature is also an important factor. At higher temperatures, lower porosities are generally observed (Maxwell, 1964). However, as both Maxwell and Fuchtbauer (1974) agree, age is the dominant factor influencing porosity. Older sediments usually show lower porosities.

The McMurray sediments are of Cretaceous age and have been buried to depths between 1000 and 1300 metres (Corbett, 1955). From Figure (3.13), porosities in the range of 35-40 percent would be expected in the fine-grained material. However, values between 23-28 percent are usually obtained (Dusseault, 1975). The reason for this is two fold. Pressure solution features and authigenic quartz overgrowths are present in the sediments. Of the two, the latter seems to predominate. In fact, Williams (1963) comments that the authigenic quartz overgrowths are almost universally present and it imparts a pseudo-angularity to the rounded grains.

Observation of overgrowths is difficult in thin section if plane polarized light is used for illumination (see before).

Solution effects are also apparent in that quartz grains show etching, pitting and frosting (compare S.E.M. photomicrographs). Williams is of the opinion that these solution effects occurred after the development of the authigenic overgrowths. However, this is contrary to the view held by Dapples (1959), in which he considers solution/pitting of quartz grains to occur in the initial or depositional stage, before precipitation of overgrowths. Krinsley and Donahue (1968), in the examination of surface textures of quartz grains using the S.E.M., find that characteristic diagenetic features (solution/pitting) are more common in older sediments. Considering the age of the sediment, it would therefore be more reasonable to assume that such features occurred in the initial depositional stage. However, age and depth of burial are not sufficient criteria for estimating the amount of grain degradation.

Porosities in the range of 25-33 percent are obtained in the coarser grained McMurray sediments. This value is in good agreement with those in Figure (3.13).

The St. Peter Sandstone is the most widespread North American arenaceous deposit of Ordovician age. It occupies the Upper Mississippi valley covering most of the middle east of North America. Deposition of this blanket deposit was from the northeast by migrating shore lines

(Dapples, 1955). It is a supermature sandstone resulting from its multicycle origin.

The area covered by this deposit is approximately 780,000 square kilometres. As Thiel (1935) points out, it is exceedingly variable in thickness, ranging from a thin layer of grains in eastern Wisconsin to a maximum thickness of 150 metres in Illinois.

The rock appears massive with occasional lines of bedding on freshly exposed surfaces. On eroded surfaces however, the bedding planes stand out conspicuously and appear in some localities, closely bedded.

The St. Peter is a friable sandstone consisting of mainly white, well sorted grains of quartz. Many of the grains have a pitted or frosted surface. The sandstone is poorly graded, nearly 90 percent of the sand is found to be of grades that are retained on the 0.25 to 0.135 millimetre sieves. The finer and coarser grades are present, but in very small amounts.

In general, the larger grains are more completely round than the smaller. However, very few of the grains are spherical: many are oval, kidney or egg shaped. Chemical analysis of the St. Peter shows essentially 100 percent quartz.

The porosity determined by Thiel is 28.3 percent. Heald (1956) puts the porosity in the clay free regions at

14 percent. The St. Peter Sandstone has been buried to a maximum depth of approximately 760 metres (Weyl, 1959). Plotting this value on Figure (3.13) shows an initial porosity of 37 percent, not unreasonable for a sand at such a shallow depth. Comparison with the average of the two final porosities gives a total reduction in porosity of 16 percent, or relatively, 43 percent. For this to occur, mechanisms other than mechanical compaction must be at work. Again pressure solution and the presence of secondary quartz account for this reduction. This time, pressure solution seems to be the dominant process.

However, as Thiel notes, the occurrence of secondary overgrowths undoubtedly has produced a decrease in the porosity of the formation. Heald illustrates the interlocking structure produced by pressure solution. He further exemplifies his illustrations to show the effect of clay coatings and grain size on pressure solution.

Weyl (1959) uses St. Peter Sandstone to test his theoretical model. Making certain assumptions, for example about the thickness of the solution film, temperature pressure solubility effects on quartz (no data was present at that time), he obtains results that are geologically reasonable. He also discovers that the temperature correction when applied to the St. Peter, changes the porosity very little.

Another interesting fact may be discovered when

comparing the McMurray and St. Peter Sandstones. Pressure solution appears to be more dominant in the St. Peter than the McMurray. It seems reasonable to infer therefore that age, not pressure is the major factor in the occurrence of pressure solution. However, age is not the whole story by any means.

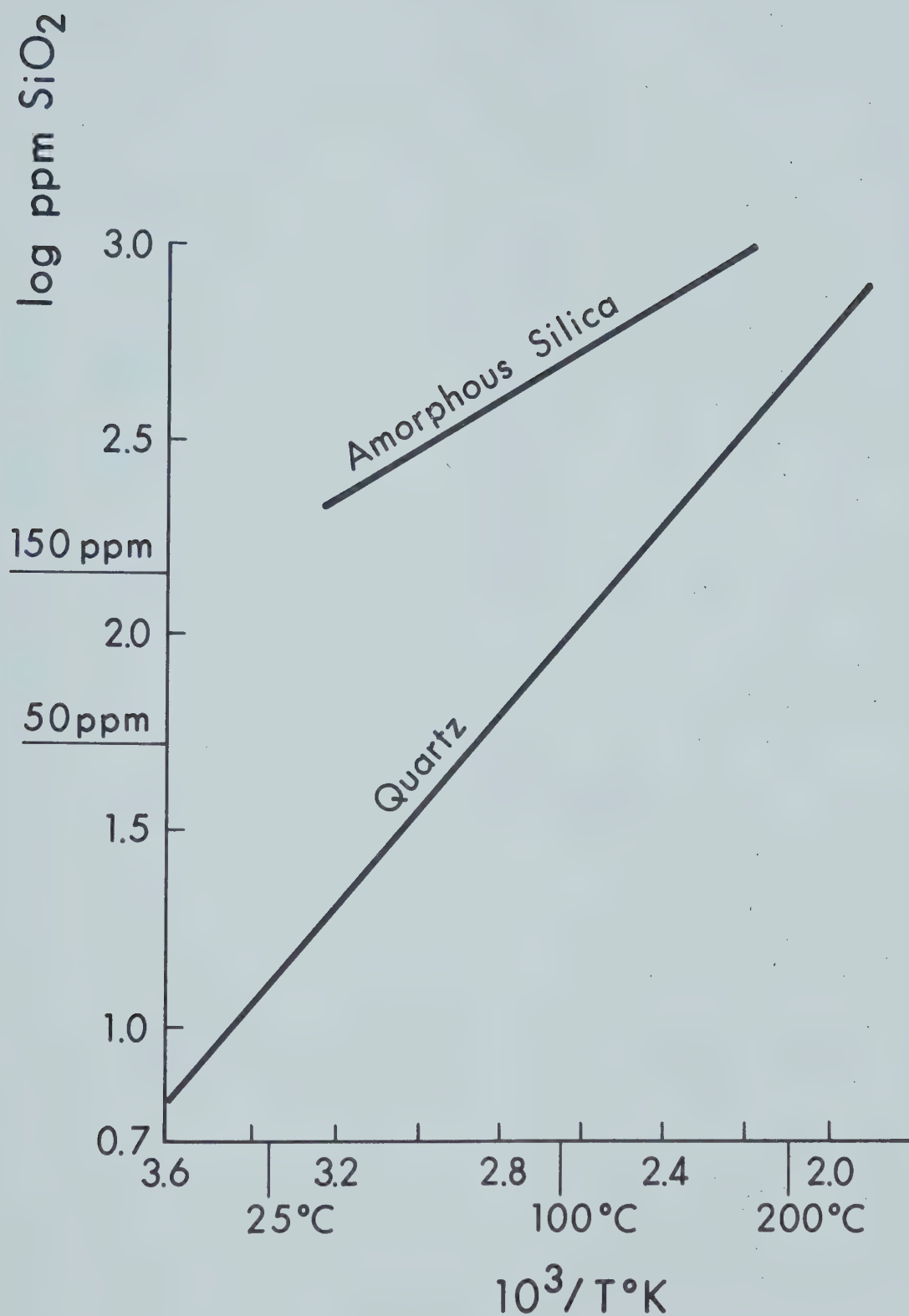


FIGURE 3.1
SOLUBILITY CHARACTERISTICS OF SiO₂
(After Siever, 1962)

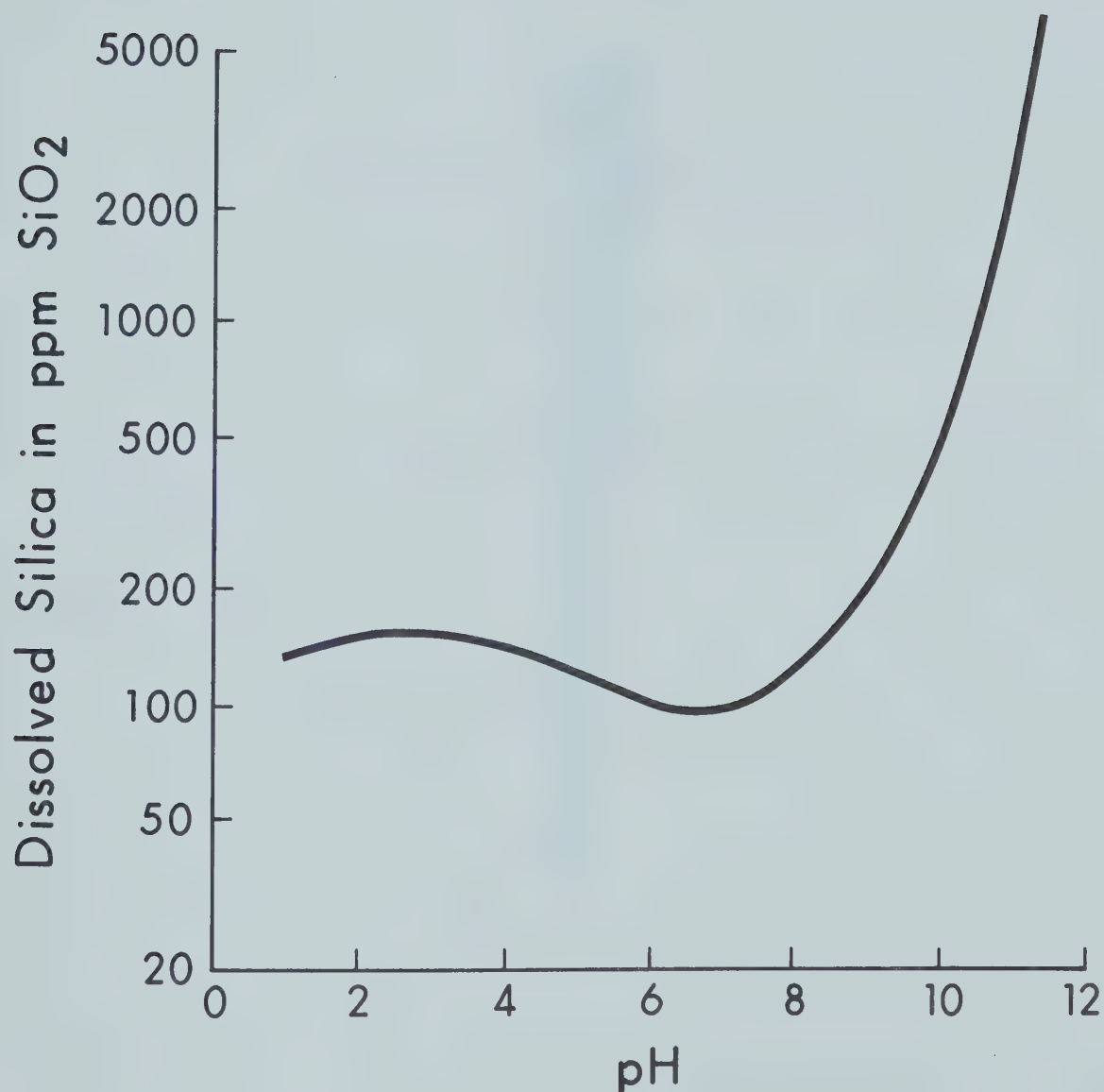


FIGURE 3.2
SOLUBILITY VARIATION OF SiO₂ WITH pH
(After Krauskopf, 1959)

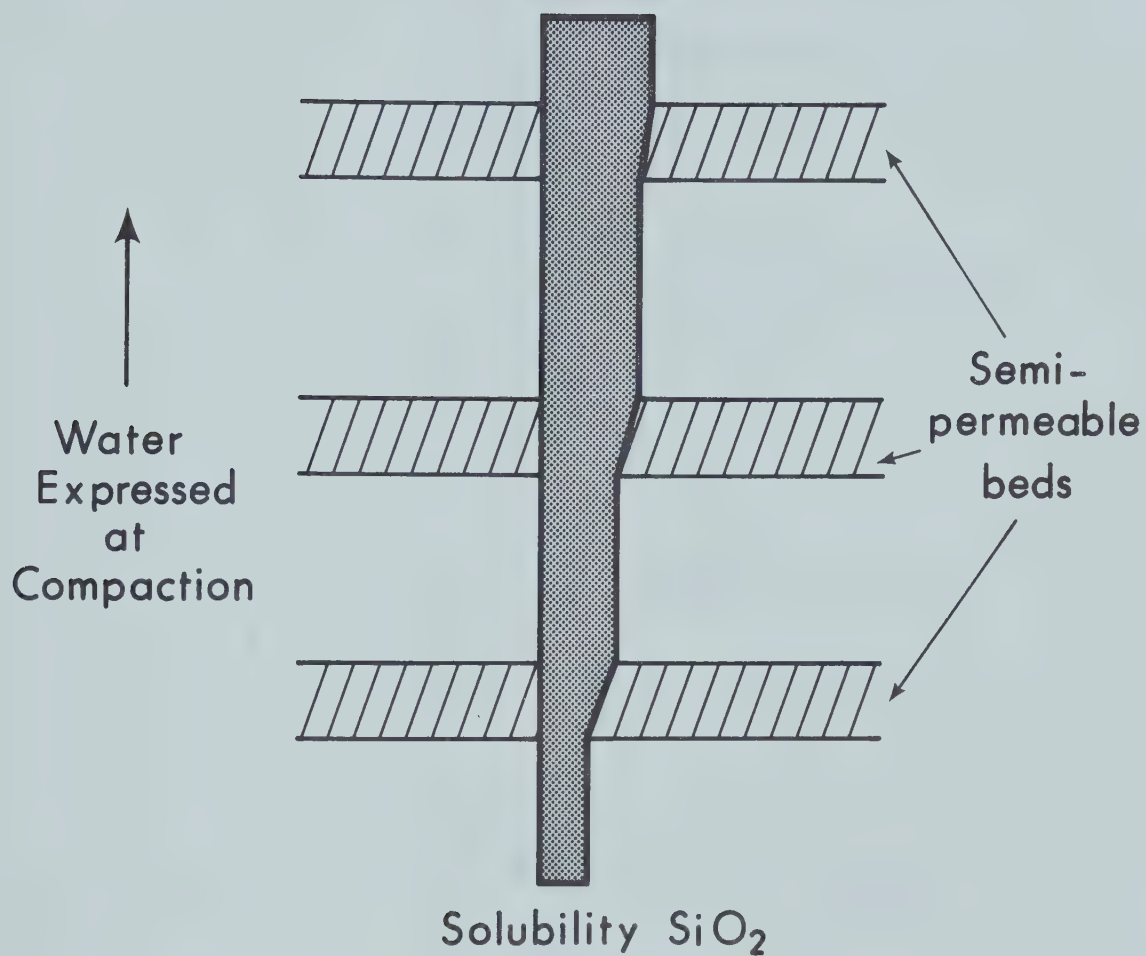


FIGURE 3.3
WATER OF COMPACTION WITH RESPECT TO SiO_2
(After Siever, 1962)

 matrix

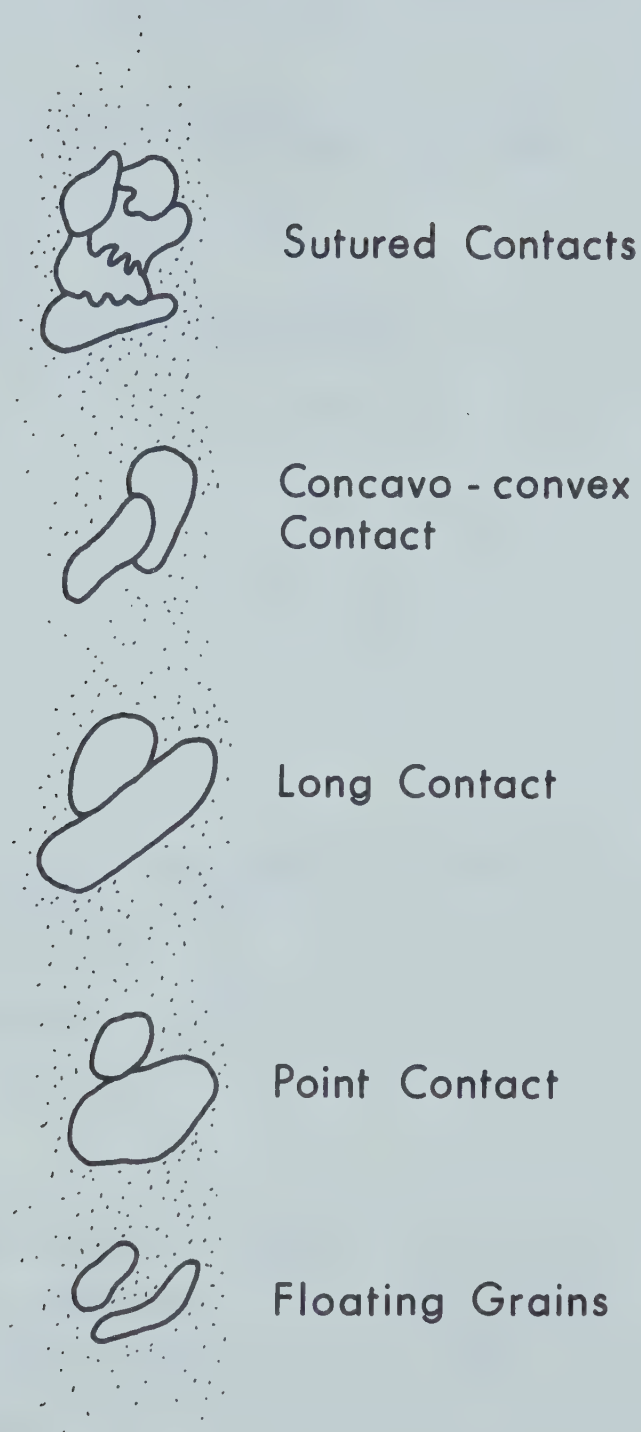
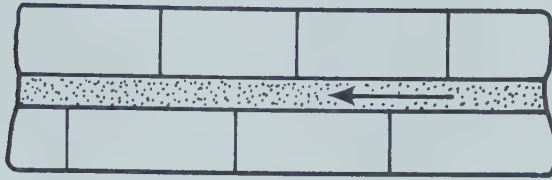


FIGURE 3.4

GRAIN CONTACTS

(After Pettijohn et al., 1973)

Thick Clay Layer, stylolites very rare



Thin Clay Layer, frequent stylolites



Enlargement showing "micro" permeability paths on alternate sides of the rock - clay interface.

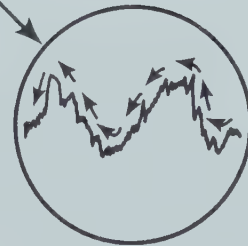


FIGURE 3.5

EFFECT OF CLAY ON STYLOLITIZATION

(After Fuchtbauer, 1972)



FIGURE 3.6
BATHURST MECHANISM
(After Weyl, 1959)

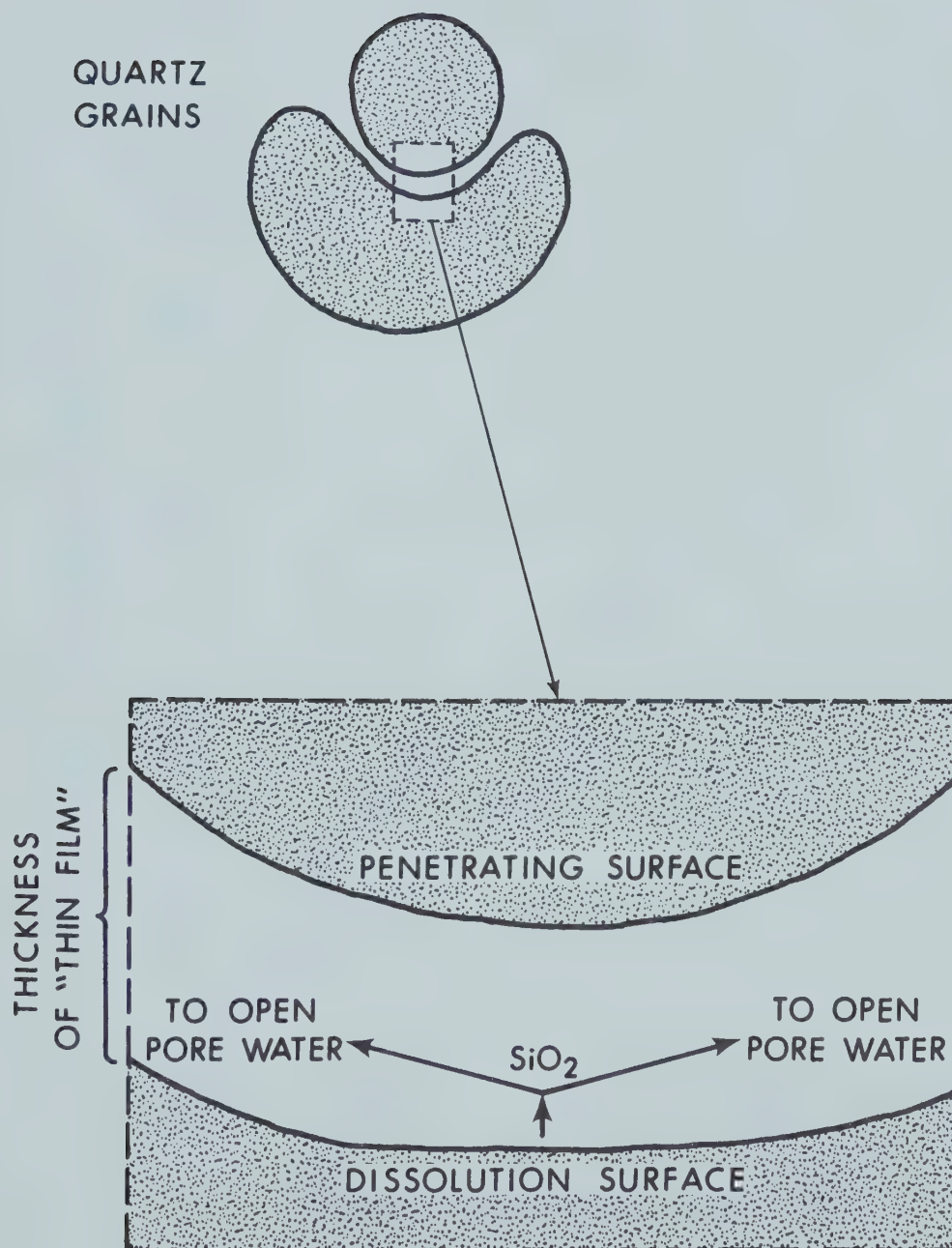


FIGURE 3.7

WEYL MECHANISM

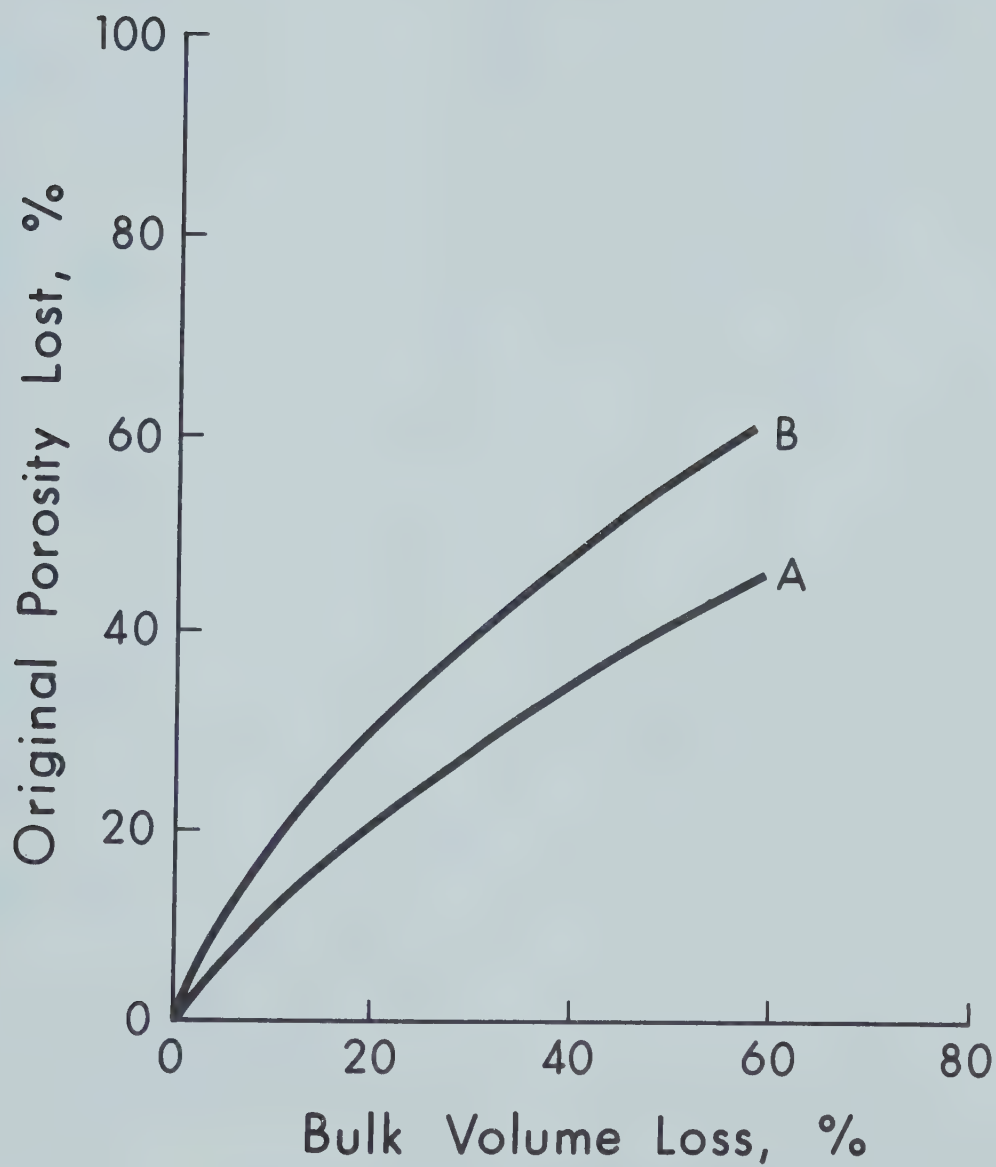


FIGURE 3.8
EFFECT OF SOLUTION TO REDUCE VOLUME
(After Rittenhouse, 1971 a)

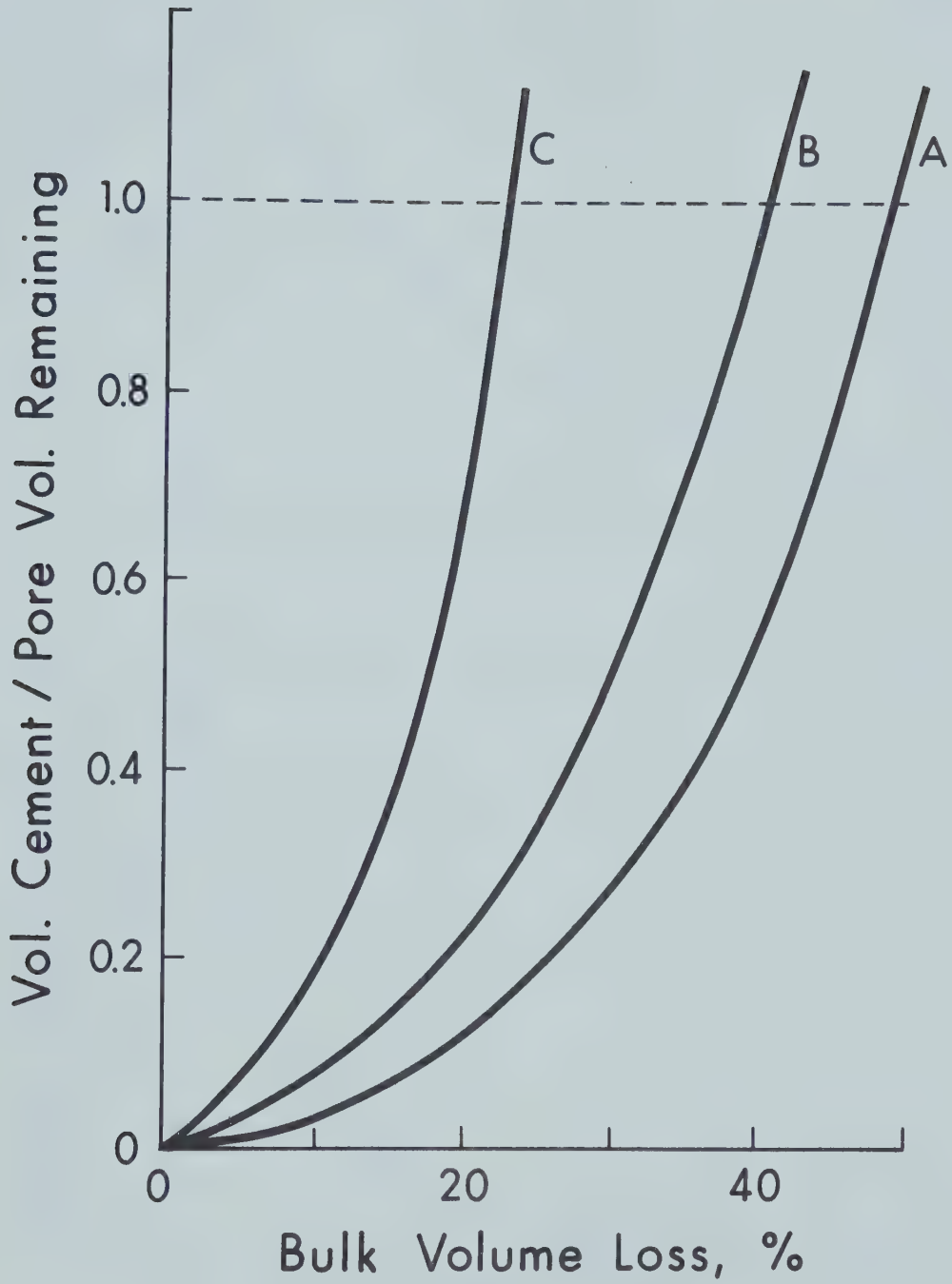


FIGURE 3.9

EFFECT OF INFILLING CEMENT TO REDUCE VOLUME

(After Coogan and Manus, 1975)

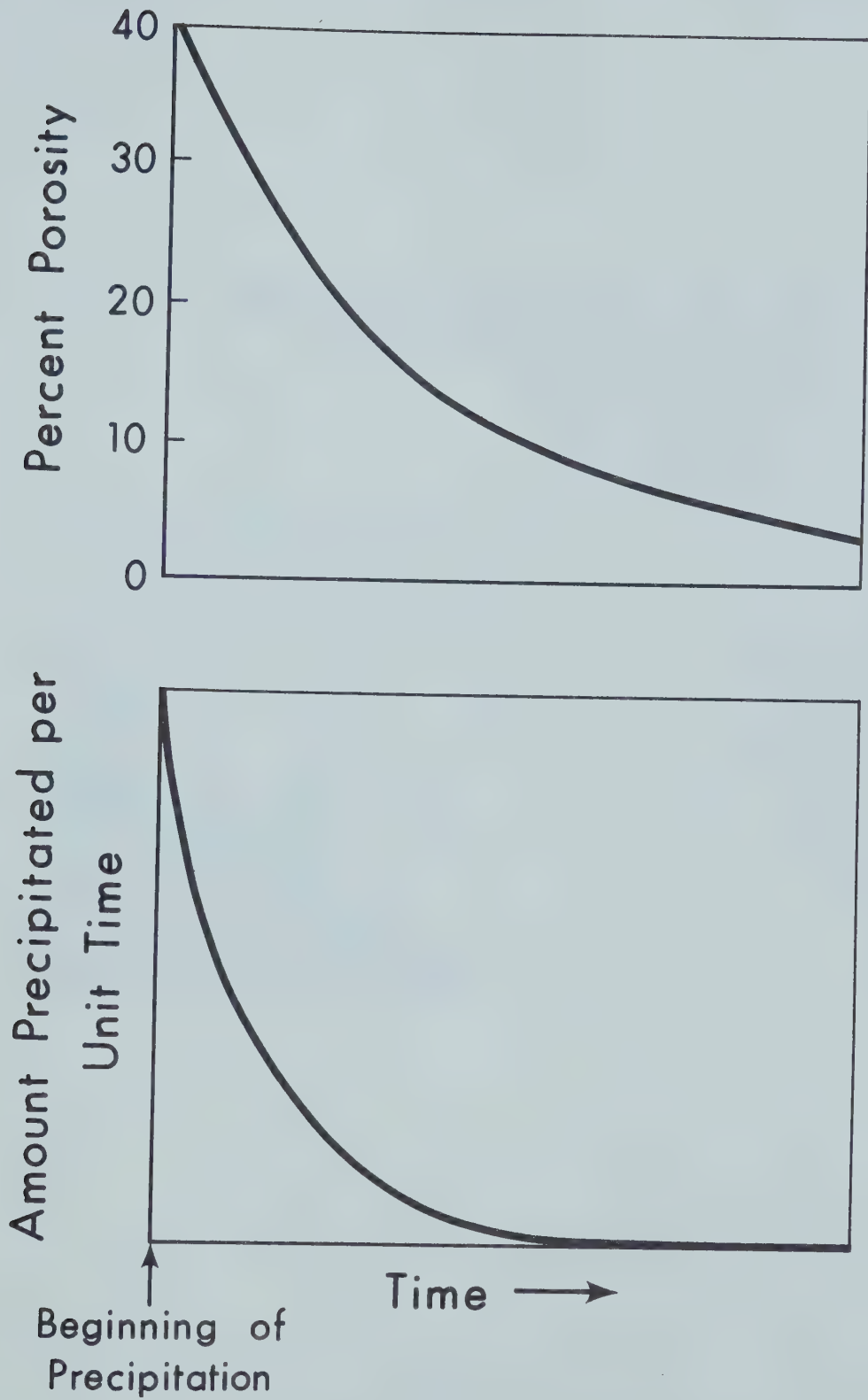


FIGURE 3.10

LITHIFICATION OF A SEDIMENT

(After Pettijohn et al., 1973)

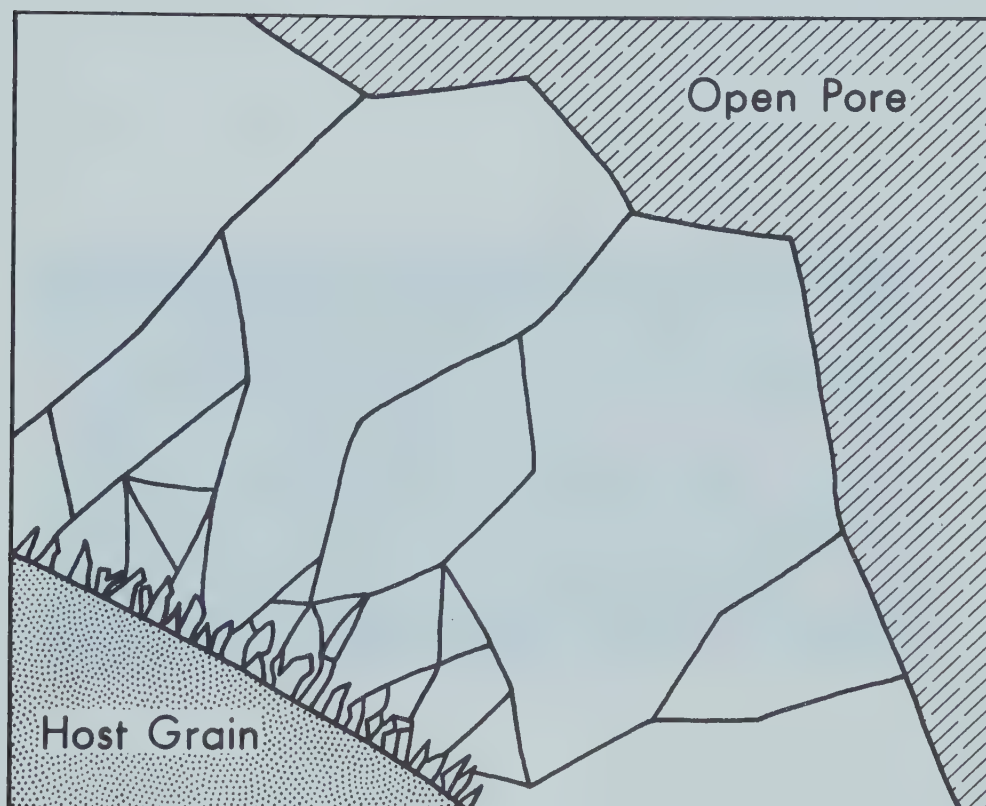


FIGURE 3.11

CRYSTAL GROWTH ON SUBSTRATE

(After Pettijohn et al., 1973)

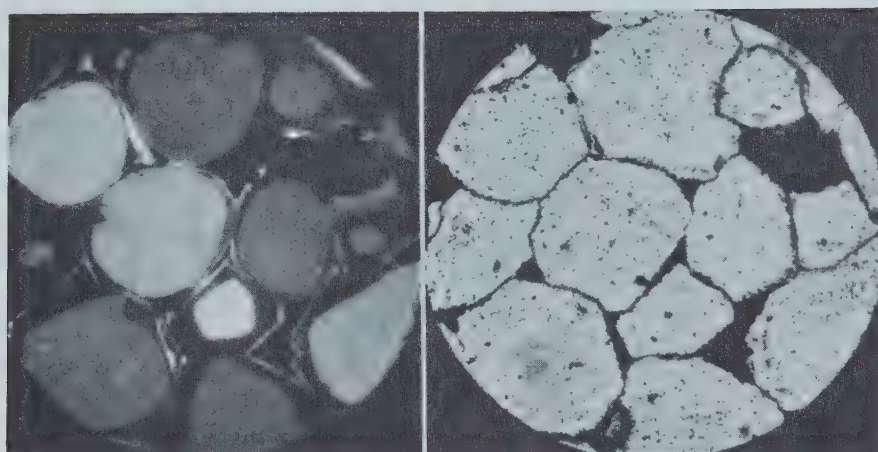


FIGURE 3.12
COMPARISON OF THIN SECTION IN PLANE-POLARIZED LIGHT
AND CATHODO-LUMINESCENCE
(After Pettijohn et al., 1973)

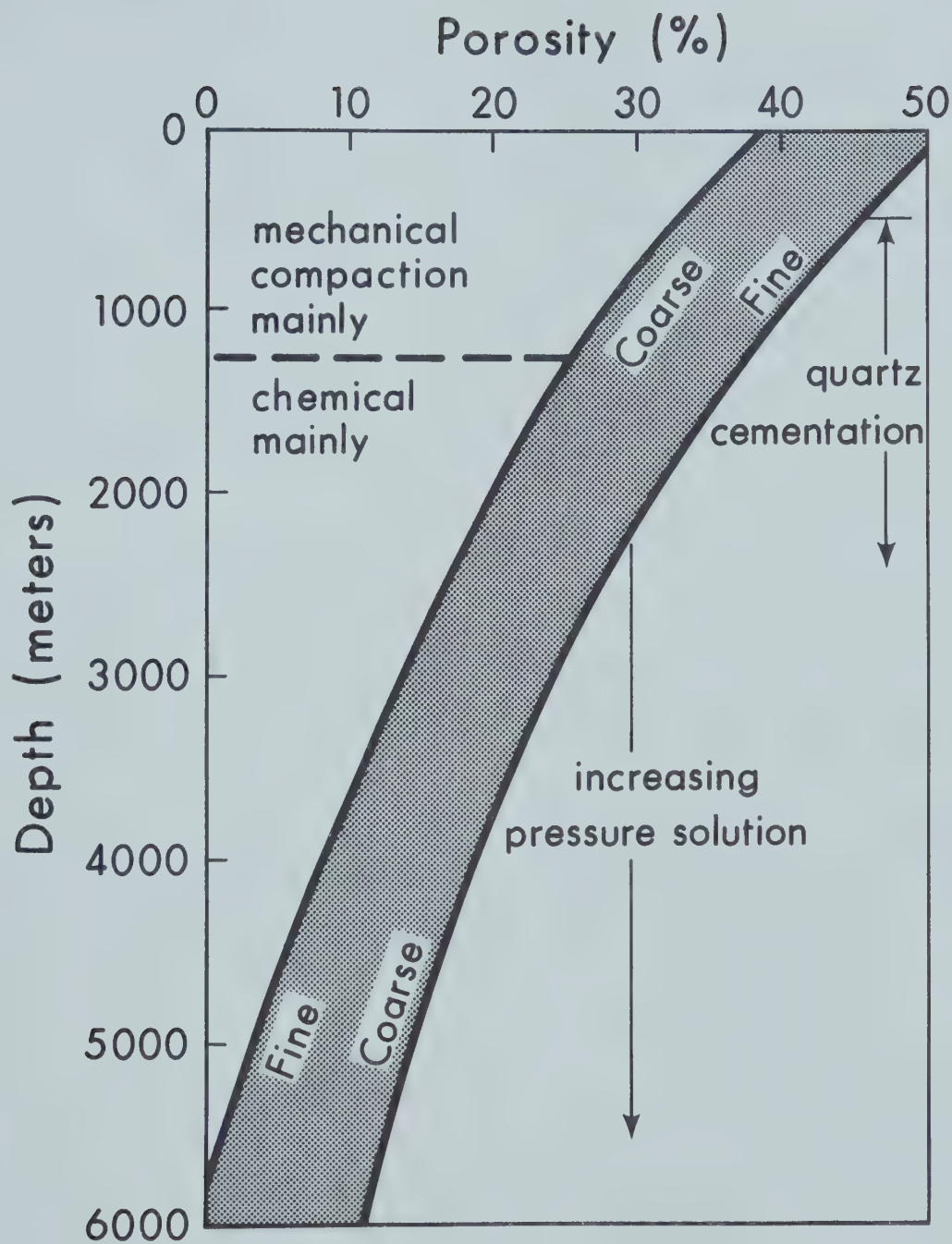


FIGURE 3.13

PREDICTION OF POROSITY WITH DEPTH FOR SANDSTONES

(After Fuchtbauer, 1974)

CHAPTER IV

EXPERIMENTAL MODEL

4.1 Introduction

This chapter presents the experiments used to simulate the structure of the friable sandstone. The first section however, explains the limitations and demands made upon past experimental work to develop pressure solution or overgrowth features. From this, it is recognized that an alternative approach must be adopted if shear characteristics are to be examined.

The following section deals with the design of the apparatus. When working with such corrosive solutions as hydrofluoric acid or concentrated sodium hydroxide, special materials must be incorporated into the overall scheme.

Finally, the experimental procedure carried out during the experiments, in an effort to establish relative consistency of structure before shear, is discussed.

4.2 Past Experimental Work

Pressure solution features for quartz have been demonstrated experimentally in the past. One of the first experiments of this kind was conducted by Siever (1962). St. Peter Sandstone was placed under pressure and heated in a reaction vessel. Petrographic examination of the grains before and after the experiment revealed no fractures. Solubility of the solution after the experiment was close to that of amorphous silica. This infers that pore waters in compacting sandstones may reach saturation with respect to amorphous silica even though quartz was originally present.

In their experimental study of sand cementation, Heald and Renton (1966), showed that with freely circulating solutions, well sorted coarse sands became cemented faster than fine-grained sands. This was due to the influx of cement into the more permeable coarse sand. Finer grained sand samples however, were cemented much more rapidly than coarse sands where the influx of cementing solutions were the same. These relationships suggest that in natural sandstones coarse-grained beds would become cemented faster than associated fine-grained beds where rapid entry of solutions occurred, as along a fault. Porosity would be greatly reduced in the coarse beds before significant porosity reduction occurred in the fine-grained beds. On the other hand, with uniform slow influx of solutions, cementation would first be completed in the fine-grained

beds. This is because quartz growth increases with specific surface of the quartz.

While studying the process of cementation, observations were made on the production of secondary quartz overgrowths. Growth rates were, among other things, dependent on the solvent concentration. Also, growth rates were greater for angular grains relative to round grains, presumably because of the greater specific surface at angular grains. Smaller angular grains were observed to grow appreciably faster than the larger grains. Also, development at overgrowths was found to be most rapid in the direction of the C-axis. These results support the views expressed in Chapter III in the section dealing with overgrowths.

Pressure solution features for quartz were further examined by Renton et al., (1969). They showed that volume reduction, through pressure solution, was much greater for fine rather than coarse-grained sand. This corroborates the view held by Fuchtbauer (1974). At high pressures (great depths), Fuchtbauer postulates that porosity reduction of fine sand is greater than that obtained for coarse sand (see Figure (3.12), Chapter III). As mentioned before, this is probably caused by the greater dissolution-reprecipitation process in fine-grained sands, relative to coarse-grained sand. This is due to the high specific surface of fine sand and tiny surface protrusions resulting from the poor rounding of such grains. Volume decrease in fine angular

sand was found to be appreciably higher than for fine rounded sand, again for the same reasons.

The experiments illustrated that solutions were necessary for pressure solution features to form. This lends credibility to the solution-film mechanisms postulated in Chapter III. Sodium hydroxide, sodium carbonate in brine solutions all produced well developed solution pits on the surfaces of the grains. With no solution present, fracture at the quartz surface at the point of grain contact occurred. Solution also occurred in distilled water, but at a rate much slower than in saline or alkaline solutions.

Renton et al. also succeeded in producing secondary growths on the grain surfaces. Commonly the largest growths were at the rims of the pressure solution pits, indicating that the solute did not move far from the site of solution. Largest growths occurred in the presence of alkaline solutions. This is probably due to the greater solubility afforded to the quartz during the pressure solution process. Simultaneous pressure solution and growth was noted early in alkaline solutions, apparently because quartz growth is very slow in nonalkaline solutions. This suggests that pressure solution could occur in natural waters of a wide range of compositions, as long as the system was open so that excessive saturation of solution would be avoided. In a closed system, the nature of the formation waters would be more critical.

It is interesting to note that Renton et al., in a summary of their experimental work, point out that;

"simultaneous pressure solution and quartz growth in sand samples produced aggregates which were considerably stronger than those resulting from cementation alone."

Competency of natural sandstones would therefore be related not only to the amount of cement but also to the extent of accompanying pressure solution or overgrowth features.

The experiments of Renton et al. and Heald and Renton, succeeded in demonstrating some of the properties of a sand aggregate having pressure solution and overgrowth features. However, the mechanism of formation was by no means natural. In Heald and Renton's experiments, pressures of over 11,000 p.s.i. combined with temperatures of 350°C were used. In fact, Renton et al. used pressures and temperatures in excess of 16,000 p.s.i. and 500°C respectively, to illustrate these properties. Such extreme conditions are necessary to speed up the solubility processes of quartz. As noted in Chapter III, the solubility of quartz is very low, approximately 10 p.p.m. at 20°C. This would mean that in order for features such as pressure solution and overgrowths to be observed naturally, long durations of time are necessary.

High temperatures and pressures necessitate the

use of special apparatus. Experiments are usually conducted using reaction vessels or "bombs", as they are commonly known. Another limitation in this kind of work is that only small amounts of sand may be tested, certainly not enough to perform a shear test.

Therefore, the effectiveness of these tests to the engineer is very limited. Not only is this due to the inadequate quantities of material resulting from such tests, but also to the high temperatures and pressures that are demanded. Tests such as these require strict controls as well if they are to be considered safe. Special metals and alloys are also necessary which increase construction costs greatly. It was therefore felt that taking everything into consideration, an alternative experimental approach should be adopted.

Thought was first given to using a soft material, for example calcite, to illustrate the strength properties characteristic of the friable sandstones. Since different mineral characteristics are evident, results may not be correlated easily to that of a sand. It was felt that the experiments should involve quartz in some manner.

Some insight to this problem was obtained from the paper by Margolis (1968). Although the article dealt primarily with electron microscopy, studies showed that diagenetic features were artificially reproduced on quartz grains by etching with hydrofluoric acid or concentrated

sodium hydroxide solutions. Etching produced random solution pits over the surface of the grain. However, it was realized, that in order for the structure of the friable sandstone to be reproduced, solution must occur at grain contacts. As pressure necessary to produce pressure solution features would not be achieved on the conventional shear box, a model which would simulate this structure was developed. This involved using glass beads of relatively different hardnesses. As the solution (HF or NaOH) passed over this assemblage, it was expected that dissolution of the softer beads would occur. This would result in the softer beads molding themselves around their harder neighbours, or conversely, the harder beads "penetrating" into the softer. In this way it was hoped that the structure of the friable sandstone, to a large extent, would be reproduced.

As may be expected with the case of such abrasive solutions, the conventional shear box was inadequate. Details of modification and other experimental apparatus are given in the following section.

4.3 Apparatus Design

Few materials are resistant against solution when exposed to hydrofluoric acid or concentrated sodium hydroxide solutions. These are extremely severe reagents. However, polytetrafluorethylene, more commonly known by its trade name Teflon, is completely inert to all forms of HF and NaOH at temperatures up to its softening point, about 300° C (Jander et al., 1971.)

As well as being inert, it has the advantage of relatively easy machining and moulding ability. Its main disadvantage however, arises from its deformation characteristics. At high stresses, for example, at those achieved on the conventional shear machine, it is slightly compressible. However, for this particular case, the advantages far outweigh the disadvantages.

The body for the circular shear box was constructed from stainless steel. It consisted of three separate parts which could be bolted together. Design followed that of the conventional shear box except dimensions were slightly larger, Figure (4.1). However, the design permitted the shear box to be used in the conventional shear box machine.

Teflon, 0.32 cms. thick, was pressure fitted to the inside of the shear box. It was also attached to horizontal parts of the shear box which move over each other

during shear. Teflon has one of the lowest coefficients of sliding friction known. Therefore, horizontal movement between these two parts occurred very easily. A rubber O-ring was placed between these parts of the shear box so when bolted down, formed a fluid-tight seal. Neoprene rings could not be used because of their reactivity with the solutions. Inert "Viton" rings were used instead.

The load cap was constructed from a solid piece of Teflon except for a metal end piece to which load could be attached. A conduit ran up the middle and out the side of the load cap. Two O-ring seals were attached to the lower end of the load cap to ensure complete fluid-tightness.

A base piece, which fitted into the shear box at the bottom, was also made from solid Teflon. It had a conduit in its upper half which could transmit fluid from inside the shear box to the outside. An appropriate connecting hole was drilled in the side of the shear box. The base piece also had double O-rings for fluid-tightness.

Porous stones were constructed from porous sheets of Teflon. It was found that an ordinary porous stone disintegrated in the presence of HF. However initially, the porous stone adjacent to the load cap proved unsatisfactory by restricting the flow of solution into the shear box. Small holes were drilled in the Teflon to alleviate the situation. They were also drilled so as to spread the solution as much as possible, since it had a central entry

point.

Connection was made from the side of the load cap to the valve by means of polyethylene tubing. From the valve, the tubing was attached to the higher reservoir. Flow of solution was by gravity alone. The tubing was also connected from the base piece to the lower reservoir. A schematic diagram illustrating the layout of the apparatus is shown in Figure (4.2). Polyethylene is not completely inert to HF. After prolonged use, it is usually found to darken due to the formation of a tar, (Jander et al., 1971). However, in these experiments the tubing preformed satisfactorily.

A Monel valve was used to adjust the rate of the solution from the reservoir. Monel is a special alloy which relies on the formation of an insoluble fluoride layer to prevent rapid corrosion of the metal due to attack by HF. It does not seem to be affected in the presence of NaOH.

After the apparatus was set up, distilled water was passed through for some time to ensure no leaks were present. The reservoir was then filled with the solution and the experiments started. These are described in detail in the following section.

4.4 Experimental Procedure

Before commencing the experiments, it was decided to examine the solution properties of the hydrofluoric acid and concentrated sodium hydroxide solutions. Small quantities of the softer glass beads were placed in polyethylene beakers containing each of these solutions. Subsequent examination of the beads after a period of several days showed that the HF solution had considerably reduced them in size. The normality of the solution was between 0.3-0.4 N. On the other hand, the NaOH solution seemed to have little effect on the beads. Gross dissolution, as observed with the HF solution, was not found. It was therefore decided that the experimental tests would be conducted with the HF as the dissolving solution. However, of the two, the HF is the more dangerous to work with. Extreme caution was exercised at all times when the solution was in use; hands and eyes were always well protected.

It was realized at an earlier stage that if the shear results of the dissolved glass beads were to be beneficial, relative consistency in conditions before shear would have to be achieved. An initial set of tests were therefore undertaken to establish a procedure to be used throughout the experiment. Some of the variables studied included flow rate of solution, percentage of different kinds of beads and pressure during dissolution. Visual

inspection of the beads after each test was made. These initial tests also afforded the opportunity of testing the reliability of the apparatus with respect to leakage. During these tests, as with the main experimental work, leakage was never a problem. However, problems were encountered due to the inadequate disposal of dissolved silica during dissolution. This was corrected in two ways: first, small holes were drilled in the porous stone to accommodate the flow more readily and secondly, a layer of 6mm diameter hard glass beads were placed in the bottom of the shear box, immediately above the porous stone. This separated the beads that were undergoing dissolution from the surface of the porous stone.

Because of gradual blocking of the porous stone from dissolved silica, and other things, the flow rate of solution could not be kept constant throughout a particular experiment. In the latter stages of the experiment, the flow rate was seen to drop considerably. It was therefore decided to measure the amount of solution passing through in terms of volume only, its rate being approximated.

The test conditions finally adopted were as follows: there was approximately an equal percentage of 4mm and 2-3mm diameter beads present in the shear box before dissolution, with a single layer of 6mm diameter beads placed on top of the bottom porous stone. The pressure at which dissolution took place was standardized at 9.2 kg/cm².

The HF solution, between 0.3-0.4 N was passed 3 times through the assemblage of glass beads from a full, 2 litre polyethylene bottle.

The 4mm beads were nearly all perfect spheres. However, the 2-3mm as well as the 6mm beads, would be more properly described as spheroid in shape. Some of these beads were grossly non-spherical and were rejected.

The usual procedure carried out during any particular experiment is summarized below.

A known weight of beads were placed in the shear box. In order to obtain the most dense configuration possible, vibration was used (Holubec and D'Appolonia, 1973). Measuring the remaining volume of shear box enabled the bulk density, hence porosity, to be calculated. However, it was realized that due to the confining effect of the sides of the shear box, this value of porosity was only an average value.

After the normal load was added, distilled water was passed through for a short time to test the equipment against leakage. Connection was then made to the HF reservoir and the solution passed through for the desired number of times. During this period of the experiment, the load cap was observed to deflect downwards, by small amounts into the shear box. After dissolution was complete, the glass beads were thoroughly rinsed with distilled water. The

new volume of the assemblage was then measured. Using the correct value of normal load, the dissolved beads were sheared under saturated conditions.

After the shear test was complete, the beads were removed from the shear box, dried and reweighed. Porosity conditions after dissolution could then be calculated. The beads were then stored away, some to be used in the photographic analyses. Some problems were encountered during shear with the beads lodging in between the two parts of the shear box. In these particular cases, the test was stopped prematurely.

In the remolded tests, the above procedure was generally followed. However, after drying and reweighing the dissolved beads, they were put back into the shear box and sheared again, at different porosities.

Table (I) summarizes the results obtained from the undisturbed shear tests. A typical stress-strain curve is given in Figure (4.3). The results from the remolded tests are given in Table (II), with a typical stress-strain curve shown in Figure (4.4). Results of dilation angle vs. horizontal displacement are shown in Figures (4.5 and 4.6).

Some of the beads were sheared under natural conditions so a comparison could be made with the dissolved tests. The results of these tests are given in Table (III). Shear was carried out in saturated conditions. As the angle

of internal friction for quartz seems to be dependent on whether saturated or non-saturated conditions are present (Procter and Barton, 1974), consistency was adopted throughout.

To compare the configuration of the assemblage before and after dissolution, thin sections were made. A synthetic Hysol resin and catalyst was used to "cement" the beads so that a thin section of the structure could be prepared.

The results of the experimental tests, and their relation to the natural oil-sand, are discussed in the following chapter. Photographic analyses involved scanning electron microscopy and thin section evaluation, and these are also discussed in detail.

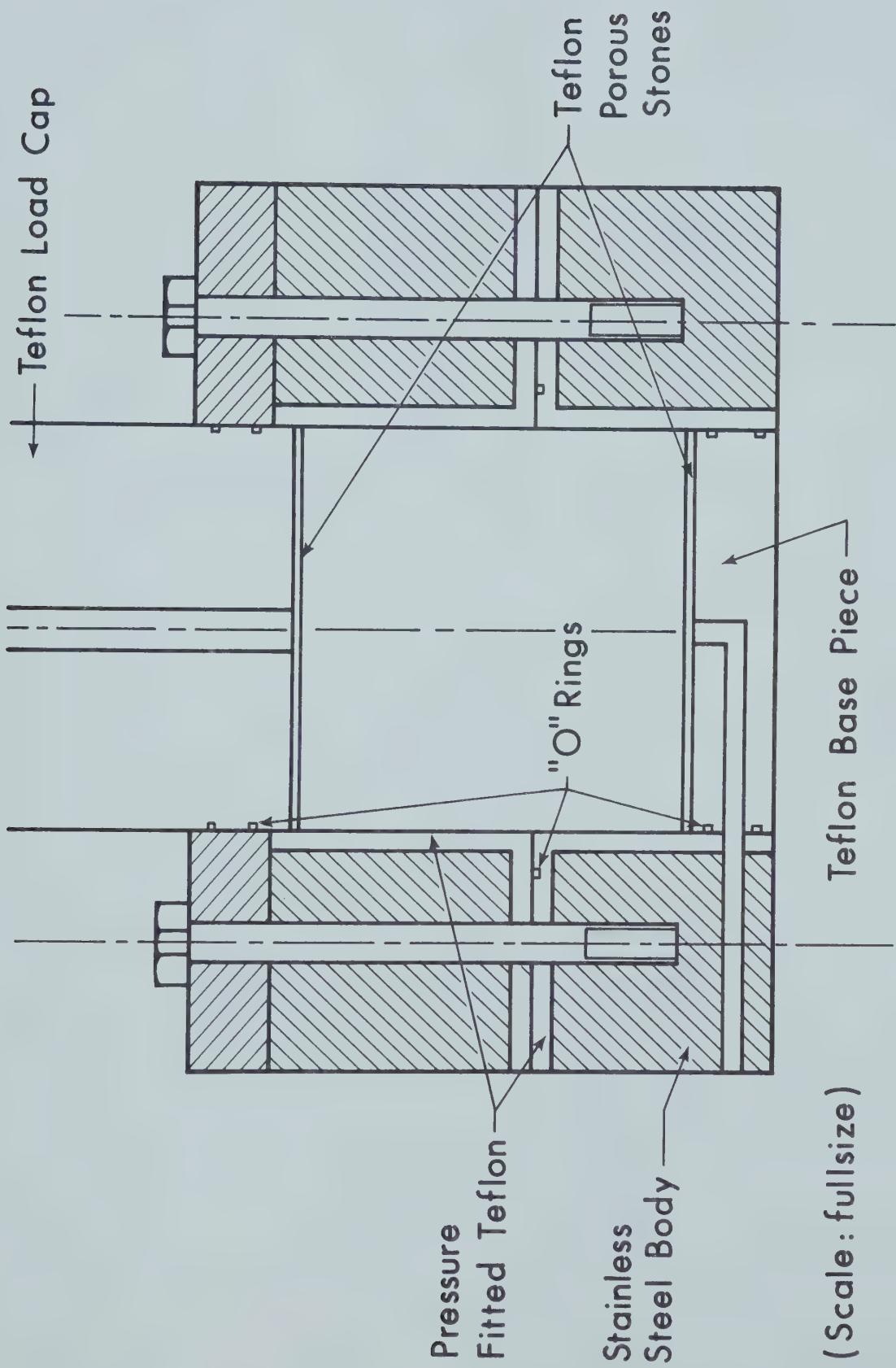


FIGURE 4.1
CROSS-SECTION OF SHEAR BOX

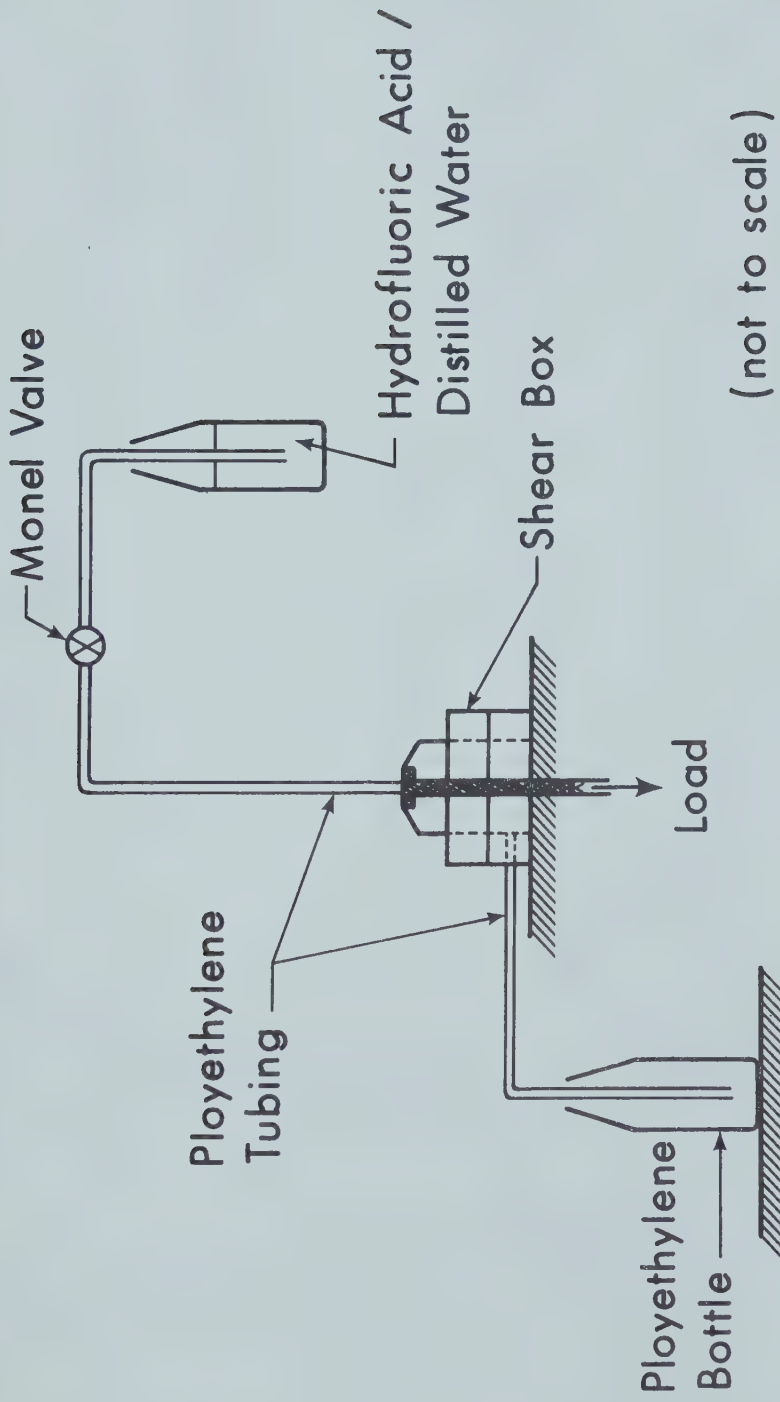


FIGURE 4.2
LAYOUT OF APPARATUS

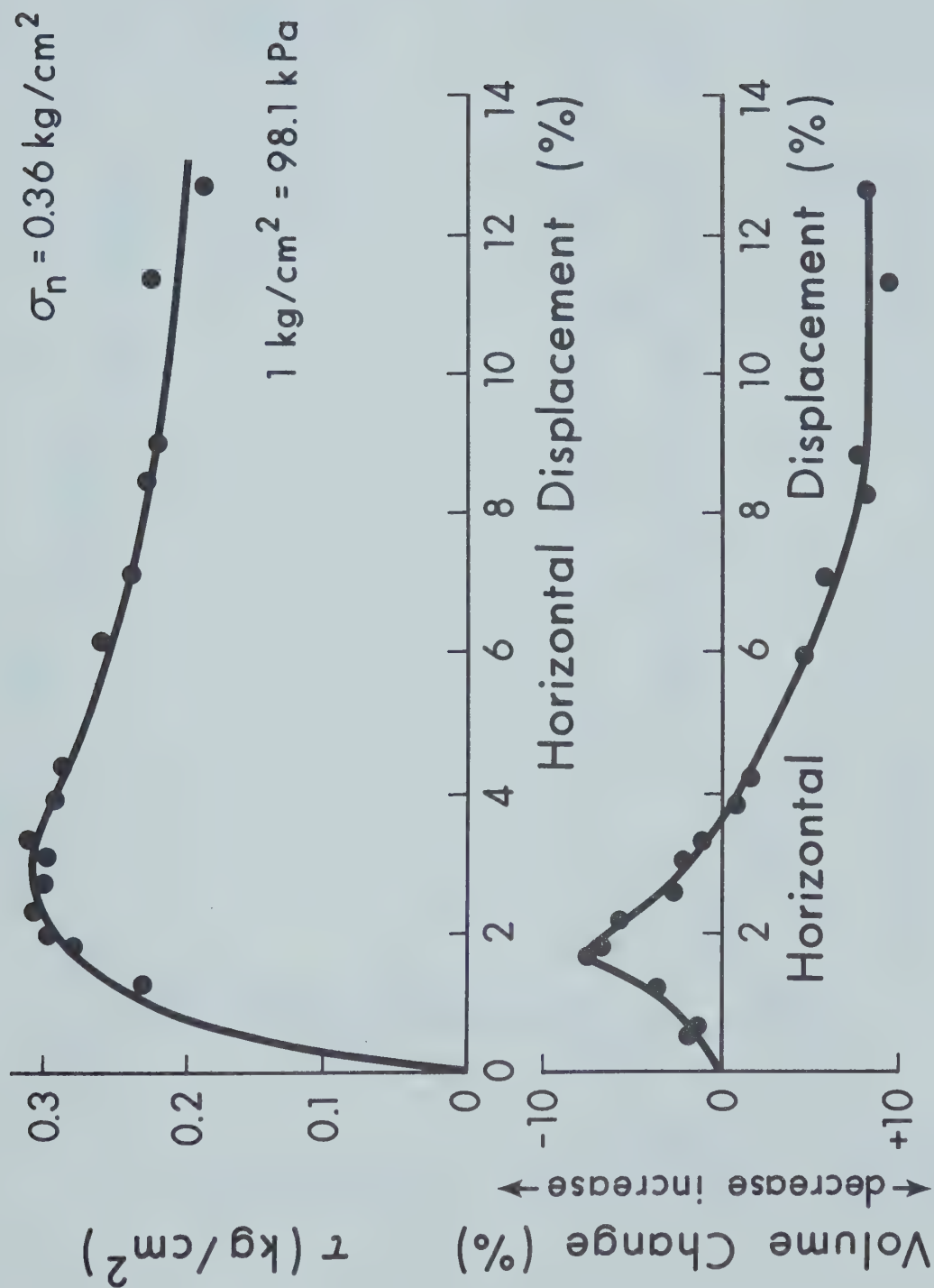


FIGURE 4.3
TYPICAL UNDISTURBED STRESS-STRAIN CURVE

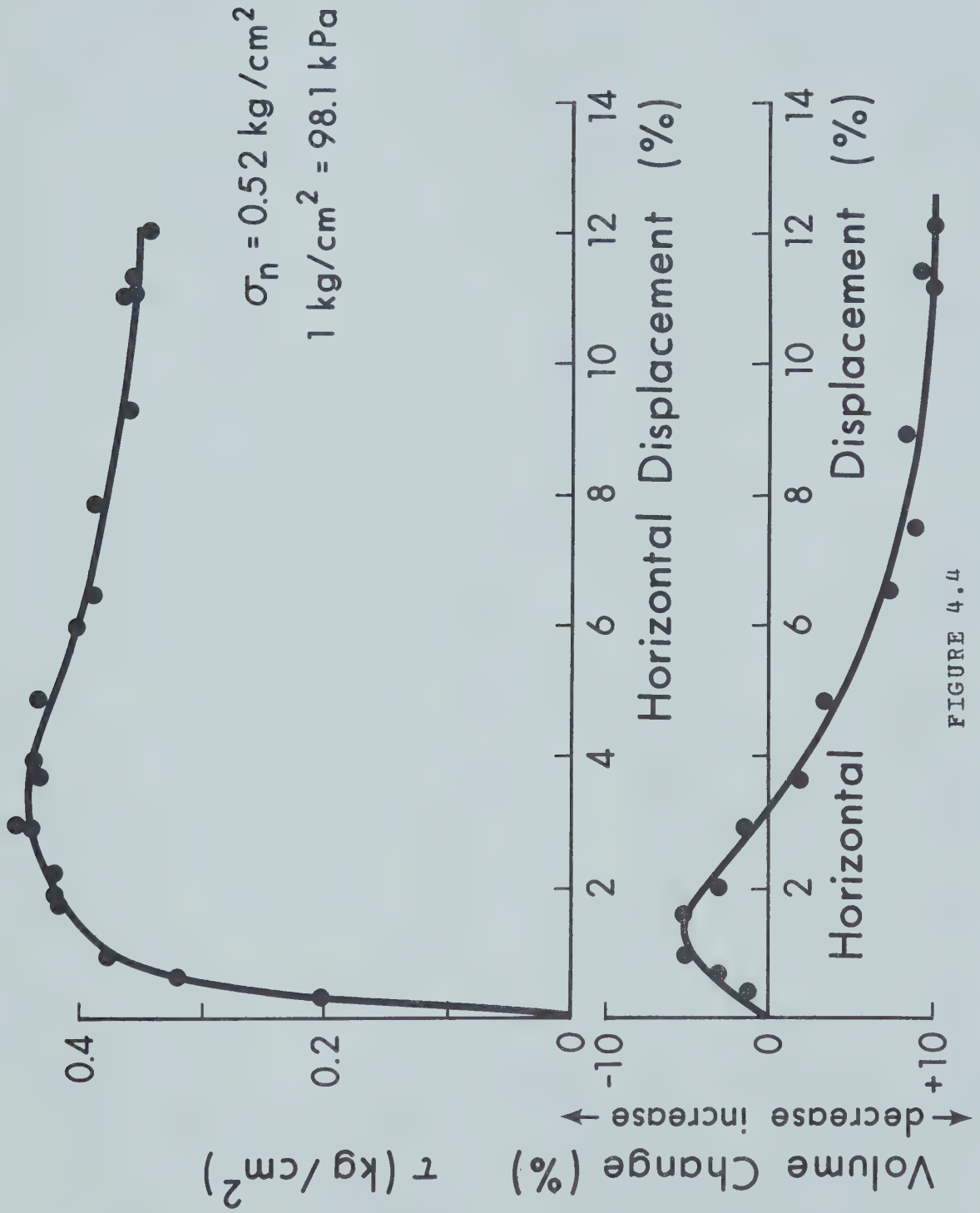


FIGURE 4.4
 TYPICAL REMOLDED STRESS-STRAIN CURVE

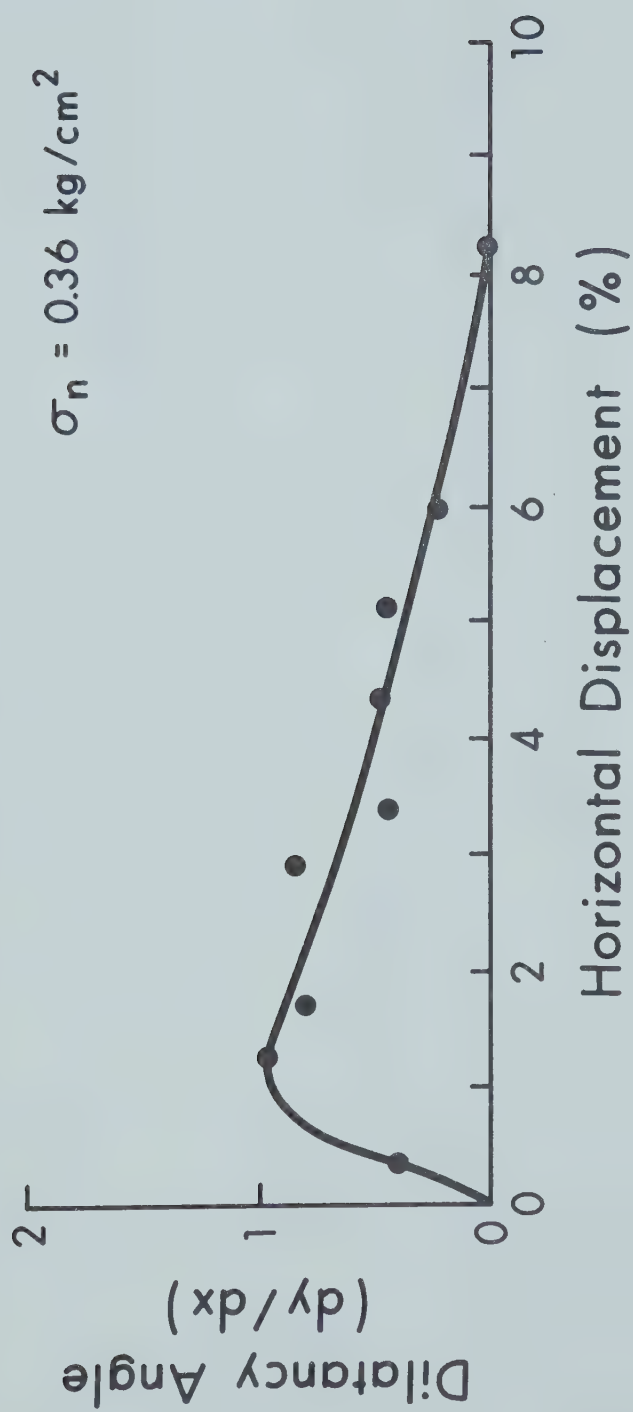


FIGURE 4.5
DILATION ANGLE (DY/DX) VS. HORIZONTAL DISPLACEMENT

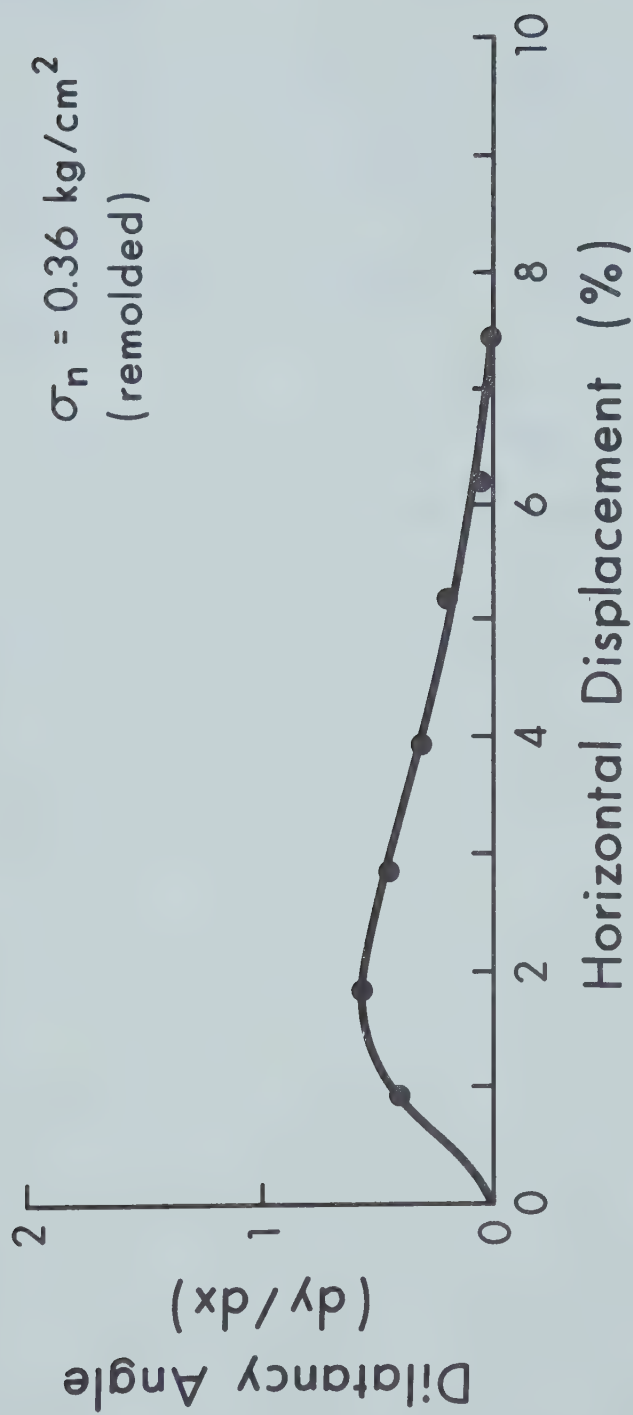


FIGURE 4.6
DILATANCY ANGLE (dy/dx) vs. HORIZONTAL DISPLACEMENT

CHAPTER V

EXPERIMENTAL RESULTS

5.1 Introduction

In this chapter, the results obtained from the experimental work are discussed. The first section presents these results and some correlation with the shear strength data for the McMurray Sandstone is made. Reasons why these results differ are given.

The following section deals with the photographic analyses (scanning electron microscopy and thin section data) of the test samples before and after dissolution. It is shown that the effect of dissolution is to produce an interlocking structure. Finally, some comments are made on the similarities between the materials studied here and randomly jointed rock.

5.2 Strength Data

From the series of tests, a curved Mohr failure envelope was obtained, Figure (5.1). At low effective normal stresses; 0.5-1.0 kg/cm², ϕ' values between 47-53 degrees were observed, while at high stresses, 7 kg/cm², a ϕ' value of 36 degrees was found, Table (I).

An average value of 33 degrees was obtained for the residual angle of friction. However, some tests were stopped prematurely and residual values were not found. This was due to the fact that at large strains, the dissolved glass beads would sometimes lodge in between the two parts of the shear box. Further straining may have damaged the Teflon so the test was terminated. This happened on five occasions.

Also plotted on Figure (5.1) are the strength results of the beads in their natural state. An average angle of friction of 33.4 degrees was observed.

First, comparing the Mohr failure envelope obtained for the natural oil sand and that of the model, a similarity is recognized. However the curvature obtained from the model tests is less pronounced; that is, strengths at lower stress levels are less. This may be due to a variety of reasons, the major ones being grain size distribution and nonuniform effect produced by the dissolving solution.

The natural material is composed of fine-grained sand, well graded compared to that of the model. As grading is increased, porosity is reduced and strength is increased. An average density of 2.25 gm/cc was obtained for the natural material whereas in the model, a density greater than 1.75 gm/cc was rarely achieved. Strength may also be size dependent. Results obtained by Marsal (1973) on crushed aggregate suggest that shear strength decreases as the grain size increases.

It was noticed (compare S.E.M. photographs) that the degree of dissolution varied among the beads. Although the beads photographed constitute a random selection, they were purposely chosen to exemplify certain features. Therefore, the foregoing statement is not completely correct. However, it is thought that concentrated paths of solution were set up within the beads. Dissolution therefore proceeded more quickly in certain areas. The interlocking effect would not be uniform across these areas and hence shear characteristics would be different. The net effect would probably be a reduction in strength.

Comparing shear characteristics before and after dissolution of the beads, a dramatic change is observed, Figure (5.1). At low effective normal stresses an increase of nearly 50 percent in ϕ' is found. For this to happen, a change in structure must have occurred. In typical stress-strain curves for the unaltered and dissolved glass beads,

the latter always showed greater initial volume increases during shear, at corresponding stress levels. This indicates that the structure is important during the shear process. Consequently, the governing strength development process at low stresses is one of greater dilatancy. At higher stresses, ϕ' reflects mineral characteristics since the mechanism of failure is one of grain fracture and shear. From Figure (5.2), maximum vertical movement at the load cap (comparable to dilation) decreases with increasing stress level. However, comparison with Figures (2.5 and 2.6) in Chapter II shows that transition in strength behaviour is not so well defined in the model material. A better correlation may seem to be drawn with Figures (5.1 and 5.3). It is also interesting to note that in the volume change (%) vs. axial strain curves, the point of maximum rate of dilation often coincides with peak shear stress, or very close to it. This is normal behaviour for a granular material.

The results of the remolded tests are shown in Figures (5.4 - 5.6). Values of ϕ' obtained by shearing the undisturbed dissolved beads are higher (except in one case), than that obtained when the beads were remolded and sheared at different porosities. The structure produced from dissolution therefore must play a significant role in the shearing process.

The exception to this occurred as shown in Figure

(5.4), where the undisturbed ϕ' was slightly less than the remolded ϕ' , Table (II). In this particular set of tests, only 4mm diameter beads were used. The effect of the solution therefore was to markedly reduce their weight. Thus the porosity was increased to a value greater than if a combination of 3mm and 4mm beads were used, as was done in the rest of the remolded tests. Shearing the compacted remolded beads produced a value of ϕ' slightly greater than the undisturbed result. However, the porosity at which this occurred was over 25 percent less than the undisturbed result. In Figure (5.7), a comparison was made between the undisturbed and remolded values of vertical deflection (comparable to dilation) of the load cap during shear. As porosity was reduced in the remolded tests, vertical movement increases but it is below that obtained in the undisturbed test. Typical stress-strain curves for the remolded tests show features described before. It is also seen from the remolded stress-strain results that dilation is less when compared with the undisturbed result.

Shear tests carried out on the McMurray Sandstone at immediate stress levels would yield a ϕ' angle of about 36 degrees and a cohesion of 1.5 kg/cm². Subsequent design using these strength parameters may be detrimental with regard to factor of safety. In the natural oil sand at high effective normal stresses when maximum strength is reached, there is a sudden drop to the residual value. Such behaviour is termed "brittle" and is consistent with the type of

structure inherent in the oil sand (Dusseault, 1975). This behaviour was not observed to any large extent in the model tests. One reason for this may be due to the lack of structure in the model material when compared to the natural oil sand.

Strength data and remolded results clearly indicate the influence of structure on the magnitude of ϕ' . In granular materials, structure is determined by, among other things, shape of grains, diagenetic processes and/or manner of deposition. In any case, structure exerts the main control on strength and deformation properties. As shown by Borowicka (1973), the arrangement of sand grains before shear has a direct influence upon the magnitude of the friction angle.

The effect of structure on strength has already been noted. However, since little information is available on deformation characteristics, it is thought that some correlation may be drawn from the work of Newman (1974).

Examination was made by Newman of pore volume compressibility relationships for a consolidated, unconsolidated and friable sandstone. Unfortunately he does not name the sandstones concerned. However, his results show that a consolidated sandstone behaves in an elastic manner whereas for an unconsolidated or friable sandstone, nonelastic behaviour dominates. He concludes that in the case of an unconsolidated or friable sandstone, deformation

characteristics are not merely porosity dependent and other parameters have to be considered. It may therefore be speculated that predicting deformation characteristics for friable sandstones in general, may be difficult.

Another geotechnical consideration associated with this type of grain structure is found in the appraisal of relative density. Relative density is a practical index that is used to determine the strength of a granular material.

It may be determined as:

$$Dr = \frac{e_{max} - e}{e_{max} - e_{min}} \times 100\%$$

where, e_{min} = void ratio of soil in densest condition

e_{max} = void ratio of soil in loosest condition

and e = in place void ratio

Depending on the relative density, the value of ϕ' for a sand may vary over as wide a range as 27-45 degrees (Terzaghi and Peck, 1967). Relative density may also be correlated with overburden pressure to estimate the penetration resistance of a sand (Gibbs and Holtz, 1957).

However, to obtain a value of e_{max} or e_{min} , the material must be remolded in the laboratory. With a sand that has been diagenetically altered in situ as the McMurray, correct determination of e_{max} is impossible. As noted in Chapter II, in situ densities cannot be duplicated under laboratory conditions by any combination of vibratory,

tamping or loading techniques. Therefore, recourse to relative density as a measure of strength in these type of sands is impracticable.

Characteristics of the McMurray Sandstone seem to apply equally well to the St. Peter Sandstone, another friable sandstone. Shear strength results of the latter also indicate an initial high value of ϕ' (approximately 65 degrees) which decreases at higher effective normal stresses. Triaxial tests also confirm this initial high ϕ' value. Electron microscopy reveals a dimpled grain texture indicating pressure solution phenomenon (Dusseault, 1975). It would be expected from the comments before, that deformation characteristics of the McMurray and St. Peter Sandstones would be difficult to predict.

Therefore, on the basis of these comparisons, it is tentatively suggested by the Author that friable sandstones in general have geotechnical properties which differ considerably from that of classical granular materials. The implications of this are far reaching in many facets of geotechnical engineering.

5.3 Photographic Analyses

5.3.1 Scanning Electron Microscopy

In an effort to clarify the mechanics of the shear process, scanning electron photomicrographs of the beads before and after dissolution were made.

As mentioned in Chapter IV, two types of beads were used; 4mm diameter with relatively little resistance to solution and 2-3mm diameter high resistance beads. Figures (5.8 - 5.9) show these two types before dissolution. Figure (5.10) indicates that the high resistance bead suffered little after being exposed to the solution in the shear box. However, small etch pits are visible at the top of the bead. These are probably places of crystal defects within the bead. This photograph is characteristic of the majority of high resistance beads after dissolution. The remaining photographs and subsequent discussion relate to the low resistance type.

Visual examination of the beads seemed to indicate that dissolution was concentrated in the central rather than the outer regions of the shear box. This was probably due to the initial entry of the solution being in the centre of the load cap. However, the porous stone (Teflon) was constructed to spread the solution as much as possible over the top surface. However, as already stated, the effects of solution seemed to be localized in the central regions. This supports

the view held in the preceding section that shear strength was nonuniform across the shear box.

The beads photographed constitute a random selection made from all the tests. However, they were chosen particularly so that certain features were distinct. It is believed that such features, on the whole are indicative of what was present in the shear box after dissolution.

The general effect of dissolution was to reduce the beads in size. As mentioned in Chapter IV, it was hoped that the soft beads would mold themselves around the hard beads during the dissolution process. Figures (5.11 - 5.14) show the best examples of this mechanism. Both photographs show profile and inclined views of different beads. These beads contain relatively deep depressions in which the harder bead is suspected of making contact. This is very similar to the ball and socket configuration described for the friable sandstones. However, the mechanisms are entirely different. Resulting shear at low effective normal stresses would cause the hard bead to ride up out of the depression. Hence an increase in dilatancy and strength of the dissolved material would be expected.

Obviously, packing arrangements and different percentages of hard/soft contacts, among other things, will influence the efficiency of the dissolution process. However, the remaining Figures (5.15 - 5.16) show that dissolution was more rapid in particular areas. If these

areas represent hard contact points, then an interlocking structure was achieved. This seems to be the case as is borne out by a comparison of the strength data before and after dissolution.

An interesting bead is shown in Figure (5.17 - 5.18). This bead shows the effects of shear. Although dissolution has taken place, the fracture surface is clearly visible. Striations on the surface indicate the direction of shear. This has probably occurred during a high strength test.

As a further clarification of the effects of dissolution, thin sections of the dissolved beads were made. These photographs are discussed in the following section.

5.3.2 Thin Section Data

To further clarify the structure present in the beads before shearing, thin sections of the undissolved and dissolved beads were made. Some trouble was encountered in their preparation, mainly due to tensile splitting of the beads and cement. However, several thin sections were made and are discussed below. The magnification of all photographs is approximately 12.0 times.

Figure (5.19) shows conditions before dissolution. Tangential contacts prevail between the beads. It is interesting to note how small an area of contact there is between the beads. Such a model is used in classical soil

mechanics concepts for granular materials (Rowe, 1962). The remaining photographs are of the beads after dissolution.

In Figure (5.20), the bead has been dissolved from all sides. The outer beads sit in depressions caused from the dissolution process. Figures (5.21 - 5.23) indicate the same type of structure, the tangential contacts have been destroyed and replaced with concavo-convex or long contacts. The latter is the type of contact prevailing in the McMurray Sandstone. With these contacts, a greater specific area of the bead is in contact with its neighbour. Consequently, shear characteristics will be different than that of the classical granular material.

As noted in Section 5.2, there was a great variation in shear characteristics before and after dissolution. From the above photographs, as well as from the electron microscopy data, the reason for this is quite clear. Dissolution has caused a remolding to occur in some of the beads, and an interlocking structure has been developed. The result of this is an enhancement of the shear and dilatancy characteristics at low normal effective stresses. At higher stresses, structure is suppressed and the shear mechanism is one of grain fracture.

An interesting contact is shown in Figure (5.24). The bead, after dissolution, shows what can only be called a stylolitic feature. The interdigitation of the columns is clearly visible. The mechanism by which this occurred is

unknown. However, to the author's knowledge, this is the first time a stylolitic contact has ever been reproduced artificially.

5.4 Correlation with Rock Properties

In rock mechanics, the behaviour of poor and closely jointed rock is of great concern. However, very little is known about the mechanical behaviour of such material. Analysis often assumes a granular material and follows traditional soil mechanics theory.

The structure of a poorly jointed rock closely resembles that of a friable sandstone, that is, it is made up of discrete units or blocks which interlock together to give stability. The cause of the jointing may be due to a variety of reasons; stress release joints, formation joints or flexural joints to name but a few. However, in comparing structure, the difference is essentially one of size.

To produce a material with a similar structure as that of randomly jointed rock, Rosengren and Jaegar (1968), heated a coarse-grained marble. Calcite, the main constituent of marble possesses anisotropic thermal expansion properties and therefore when heated, the grains separate along the grain boundaries. The resulting structure is shown in Figure (5.25). As seen, it resembles that of the friable sandstone very closely. Subsequent triaxial testing yielded a highly curved Mohr failure envelope with a cohesion intercept near 0 p.s.i. At low values of confining pressure a ϕ' of around 65 degrees was obtained. These are similar to the results obtained for the friable sandstone.

Some correlation may also be drawn with the properties of a crushed aggregate, since the structure is similar. Invariably shear box tests produce a curved Mohr failure envelope with high values of ϕ' at low effective normal stresses (Silvestri, 1961, Marsal, 1973). In the tests carried out by Silvestri, a crushed aggregate with a porosity of 26 percent under a load of 9 kg/cm² gave a ϕ' -value of 65 degrees.

As pointed out by Terzaghi (1962), fracture surfaces in randomly jointed rock are always located close to the slope. An exception to this however, is where the slide has a deep-seated origin. However, even with heights of 300 metres or more, values of effective normal pressure do not approach 9 kg/cm². Some natural slopes of fissured rock have even been found stable at angles of 71 degrees or more (Terzaghi, 1962). This of course assumes that seepage pressures are not present.

Traditional methods of analysis consider a randomly jointed rock as if it were a granular material. A straight line failure envelope, with a slope between 35-45 degrees, is thus assumed. From the above comments, it is seen that this grossly underestimates the strength of the material at low effective normal stresses. The resulting engineering design will therefore be overconservative.

Patton (1966), developed a theoretical framework whereby the effects of discontinuities within a rock mass

were taken into consideration. The result was a failure envelope approximated by two intersecting straight lines, the latter at a considerably lesser slope than the initial. However, as Patton points out, "failure envelopes for [rock] specimens with irregular failure surfaces are curved, and the change in slope of the failure envelope reflects in the mode of failure." In a randomly jointed rock the failure surface would indeed be irregular; also, the process of failure invariably shows initial high dilatancy (Wawersik and Johnson, 1973).

One interesting facet of the dilatancy behaviour occurs when the rock is saturated. If the time of initial deformation is too short to allow drainage, the tendency to dilate will induce suction in the pore fluid. This suction augments the effective normal stress on the fissured surfaces and dilatantly hardens the rock against further deformation. This is one of the reasons why initial rock cuts in saturated fissured material appear stable, but then after drainage has occurred, fail (Rice, 1975).

To examine further the correlation between a friable sandstone and randomly jointed rock, model studies relating to the latter may be pursued. The purpose of model studies is to observe certain characteristics in the model, and relate them to the behaviour of jointed rock masses. Shear box tests on models show that a curved Mohr failure envelope is obtained (Einstein and Hirschfeld, 1973). These

results indicate that among things, deformability increases with decreasing "joint" spacing. Also, a distinct transition from brittle to ductile behaviour may be recognized depending on stress level. This transition is also affected by "joint" spacing in the model. Therefore, it may be concluded that model studies of jointed rock masses show characteristics similar to that already described for some friable sandstones.

In conclusion, the correlation between the properties of a friable sandstone (structure, strength and deformation behaviour) and that of a randomly jointed rock is evident. Present methods of analysis for the latter often assume a conventional granular material, which may result in an underestimate of strength properties. A better representation of its behaviour would be expected if a curved Mohr failure envelope was used.

Crystalline rocks (for example, granite, marble and quartzite), consist of more or less irregularly shaped particles which fit together like blocks between the joints in a randomly jointed rock. The macrostructure of the jointed rock is a large scale model of the microstructure of the intact rock. Noting the presence of microcavities between the particles (Sprunt and Brace, 1974), and the decrease in ϕ' with increasing stress level during shear box tests (Terzaghi, 1962), it is interesting to speculate whether a curved Mohr failure envelope would not be a better

representation of failure criterion in crystalline rocks also.

TEST NUMBER	n (%) (initial)	n (%) (final)	σ_n (kg/cm ²)	ϕ_p' (degrees)	ϕ_r' (degrees)
8	40.47	34.21	0.49	43.2	-
9	35.02	37.90	0.33	45.2	31.5
10	34.43	40.83	0.36	44.5	31.0
12	38.08	42.37	0.52	53.5	-
14	41.47	44.98	0.52	43.1	-
20	36.00	-	0.33	43.7	33.8
21	38.08	40.41	0.45	46.6	35.5
26	29.28	40.96	2.03	40.8	33.0
30	30.95	34.23	3.61	39.6	30.7
31	29.74	-	5.19	38.5	35.2
32	29.81	33.17	5.19	41.2	30.8
33	30.18	33.06	5.19	38.7	32.5
34	30.18	34.68	6.75	39.5	33.4
35	31.70	31.70	7.33	35.7	31.8
36	31.70	32.07	8.75	35.7	-
37	29.85	32.81	10.54	35.3	30.7

TABLE I. UNDISTURBED SHEAR RESULTS

TEST NUMBER	n (%)	σ_h (kg/cm ²)	ϕ_p' (degrees)	ϕ_r' (degrees)
14	44.98	0.52	43.1	-
16	40.48	0.52	40.5	32.0
17	36.80	0.52	43.2	31.2
18	39.95	0.52	40.4	31.5
19	37.13	0.52	41.1	32.0
21	40.41	0.45	46.6	35.5
22	42.94	0.45	38.2	32.5
23	40.19	0.45	38.5	32.1
24	40.01	0.45	37.6	32.8
25	39.74	0.45	41.4	33.1
26	40.96	2.03	40.8	33.0
27	41.84	2.03	34.0	32.5
28	41.39	2.03	35.8	32.0
29	39.93	2.03	37.0	33.0
30-1	43.58	2.03	35.4	31.2

TABLE II. REMOLDED SHEAR RESULTS

TEST NUMBER	n (%)	σ_h (kg/cm ²)	ϕ_p' (degrees)	ϕ_r' (degrees)
13	38.1	0.52	33.4	31.2
15	38.5	1.20	34.4	31.0
38	38.3	10.54	35.2	32.1

TABLE III. NATURAL SHEAR RESULTS

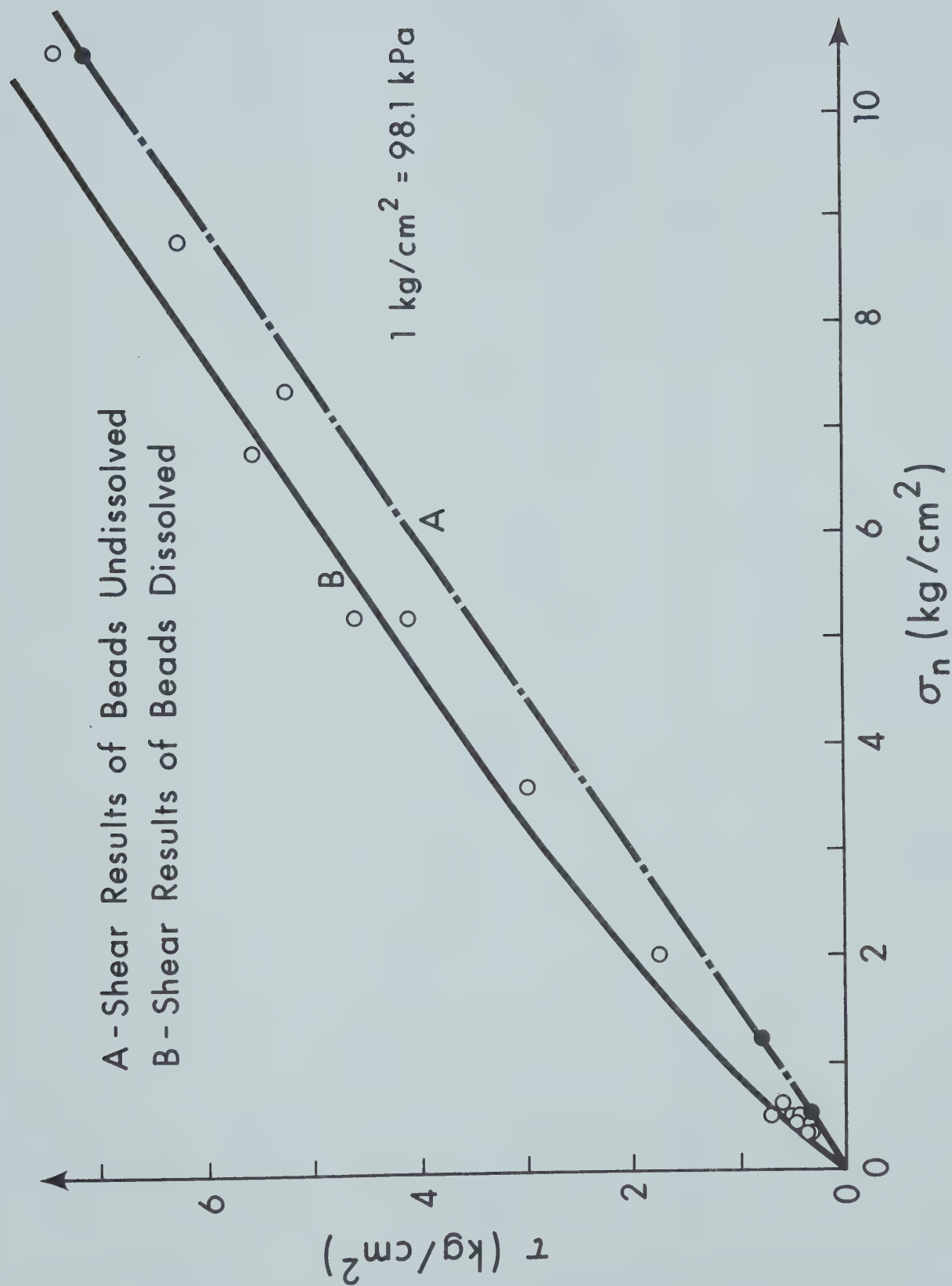


FIGURE 5.1

SHEAR CHARACTERISTICS BEFORE AND AFTER DISSOLUTION

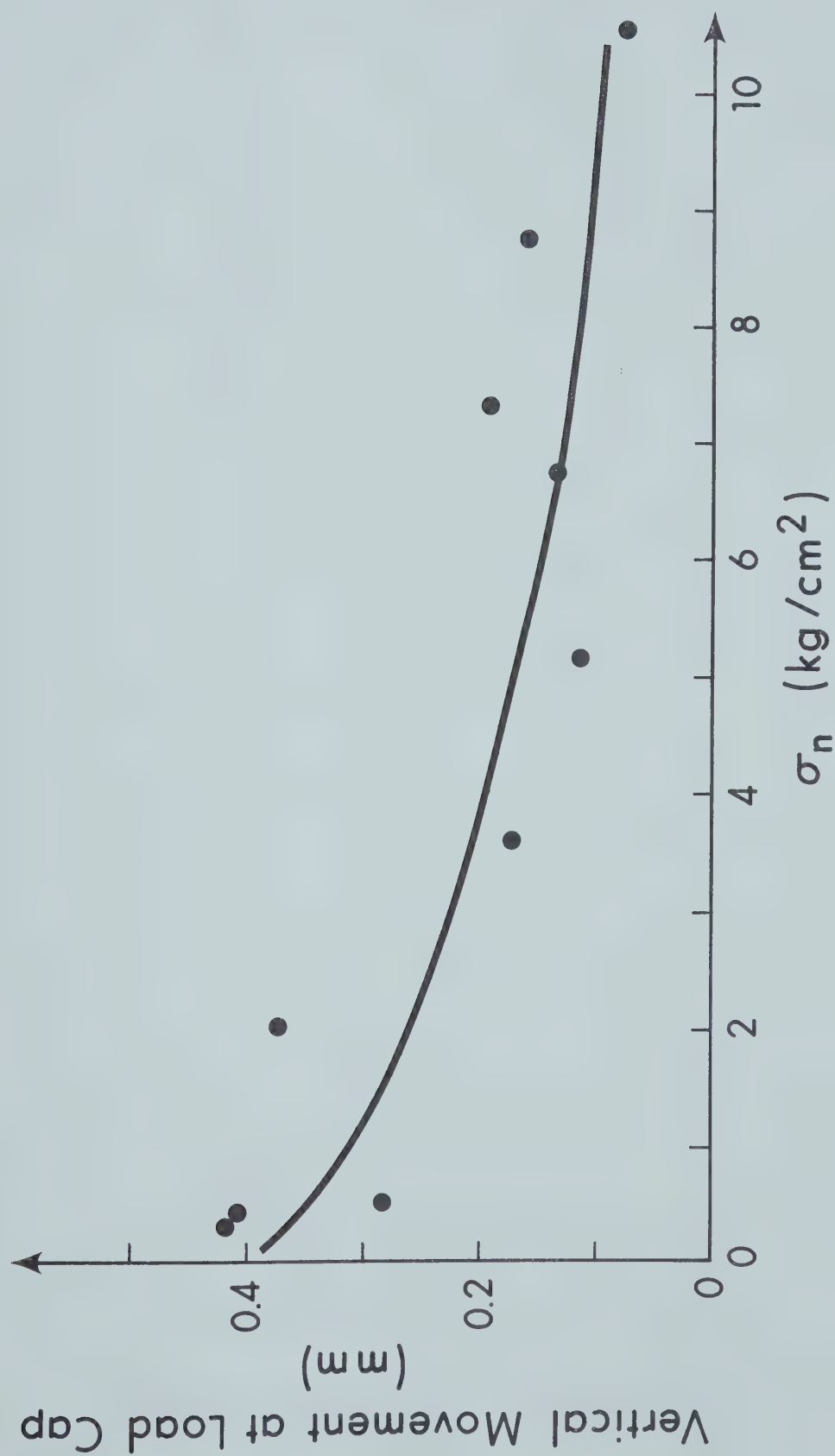


FIGURE 5.2
VERTICAL MOVEMENT AT LOAD CAP

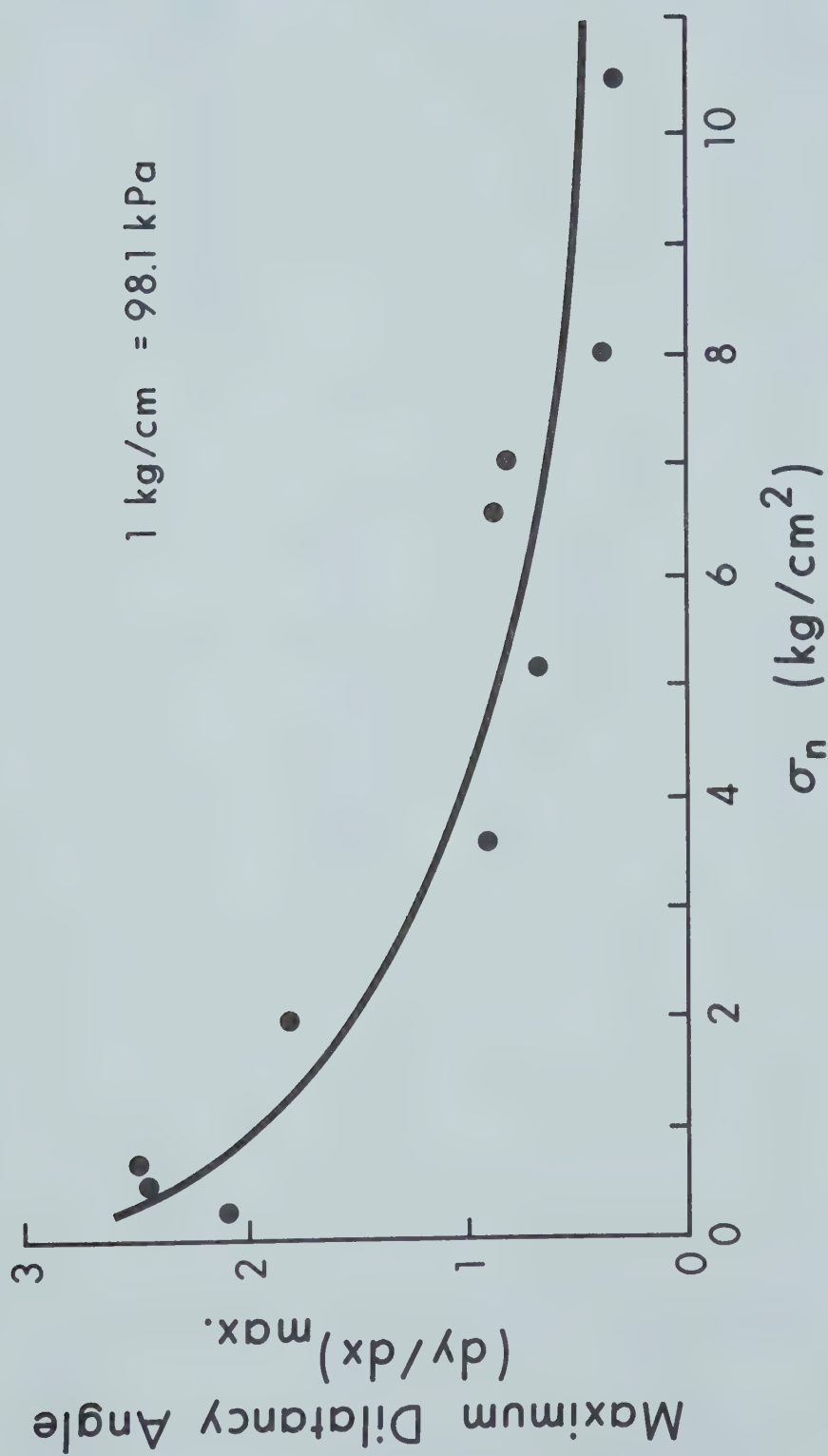


FIGURE 5.3

MAXIMUM DILATION ANGLE $(DY/DX)_{\max.}$ VS. NORMAL LOAD

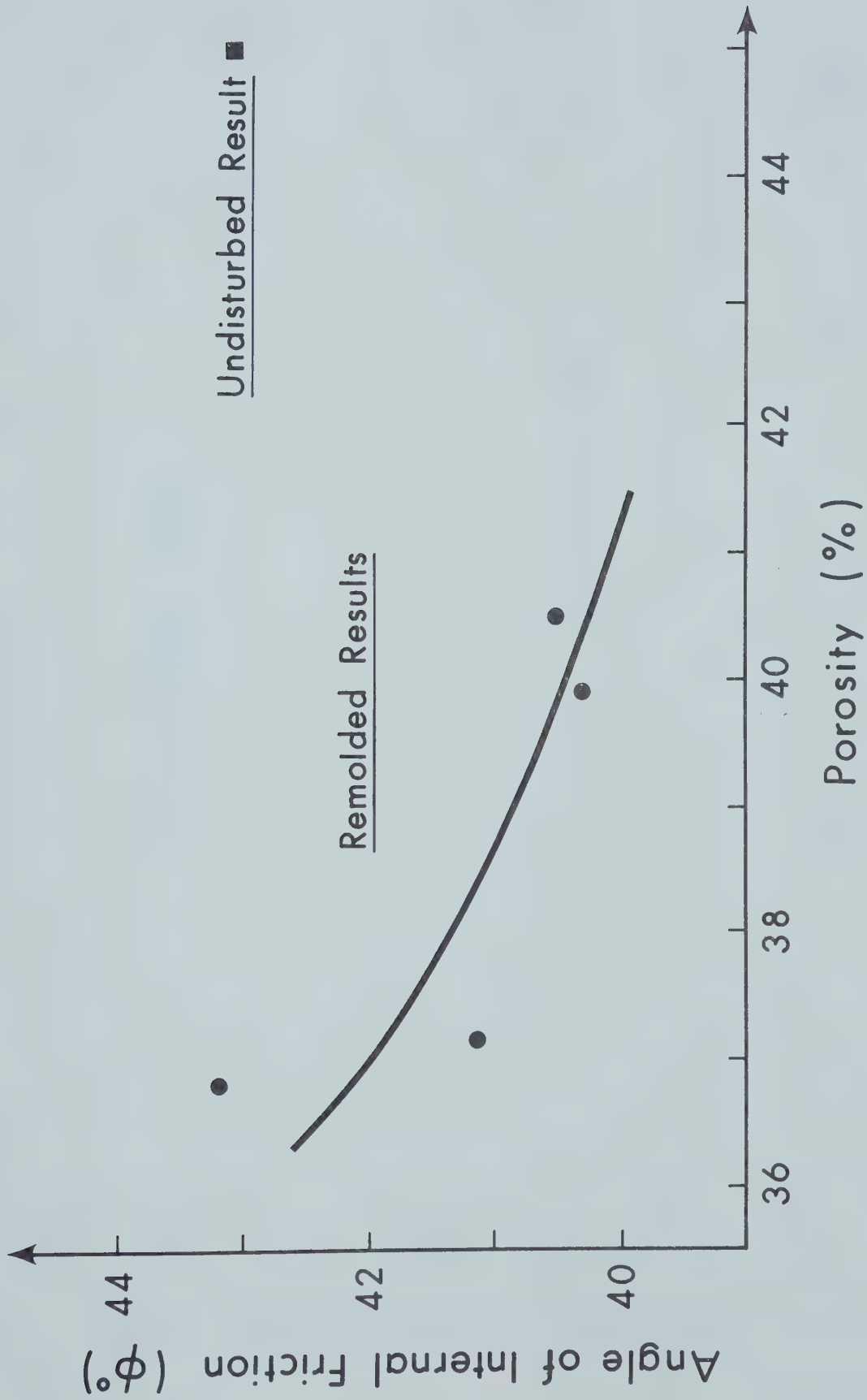


FIGURE 5.4
 REMOLDED RESULTS (POROSITY vs. ϕ°)

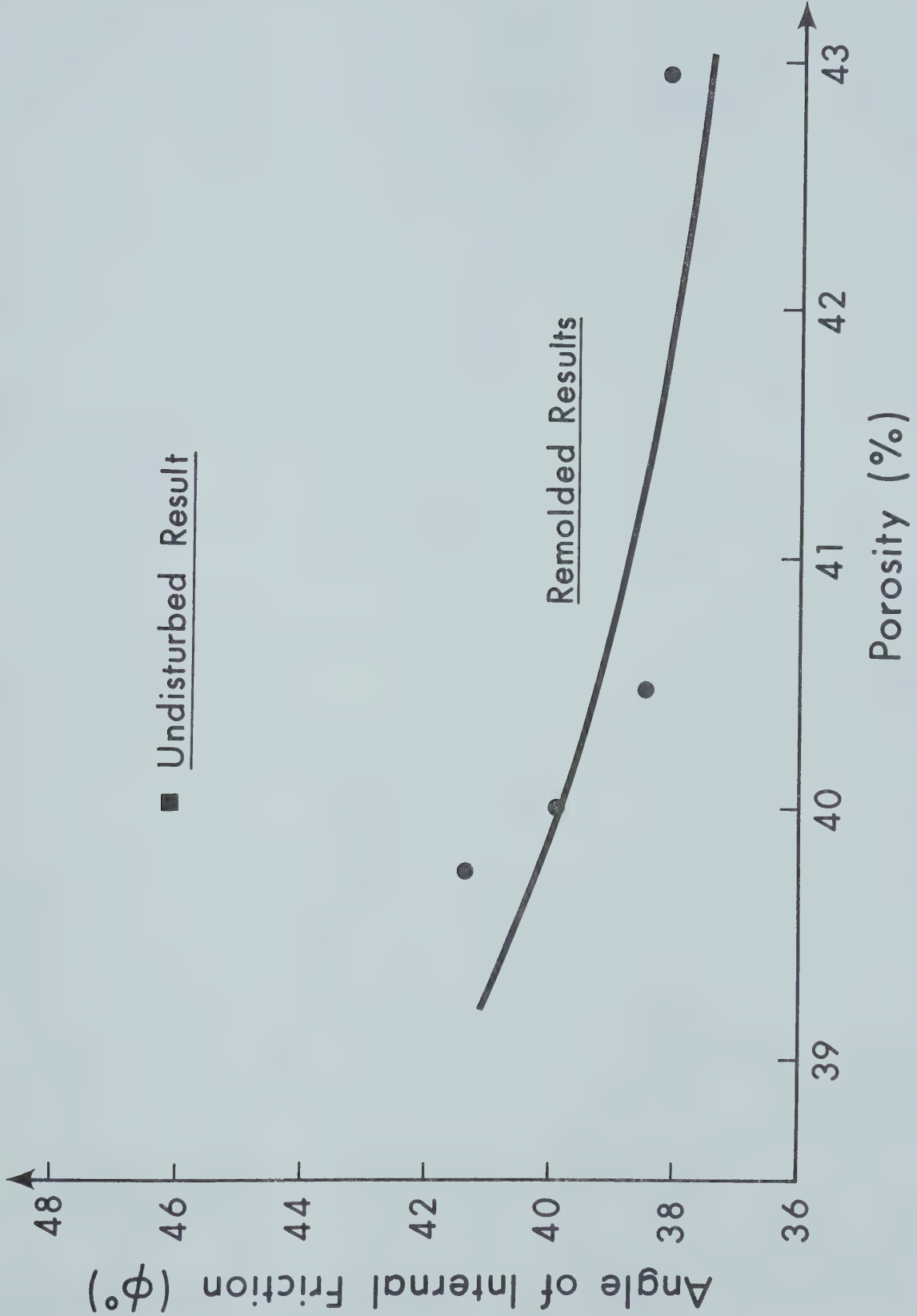


FIGURE 5.5
REMOLDED RESULTS (POROSITY vs. ϕ°)

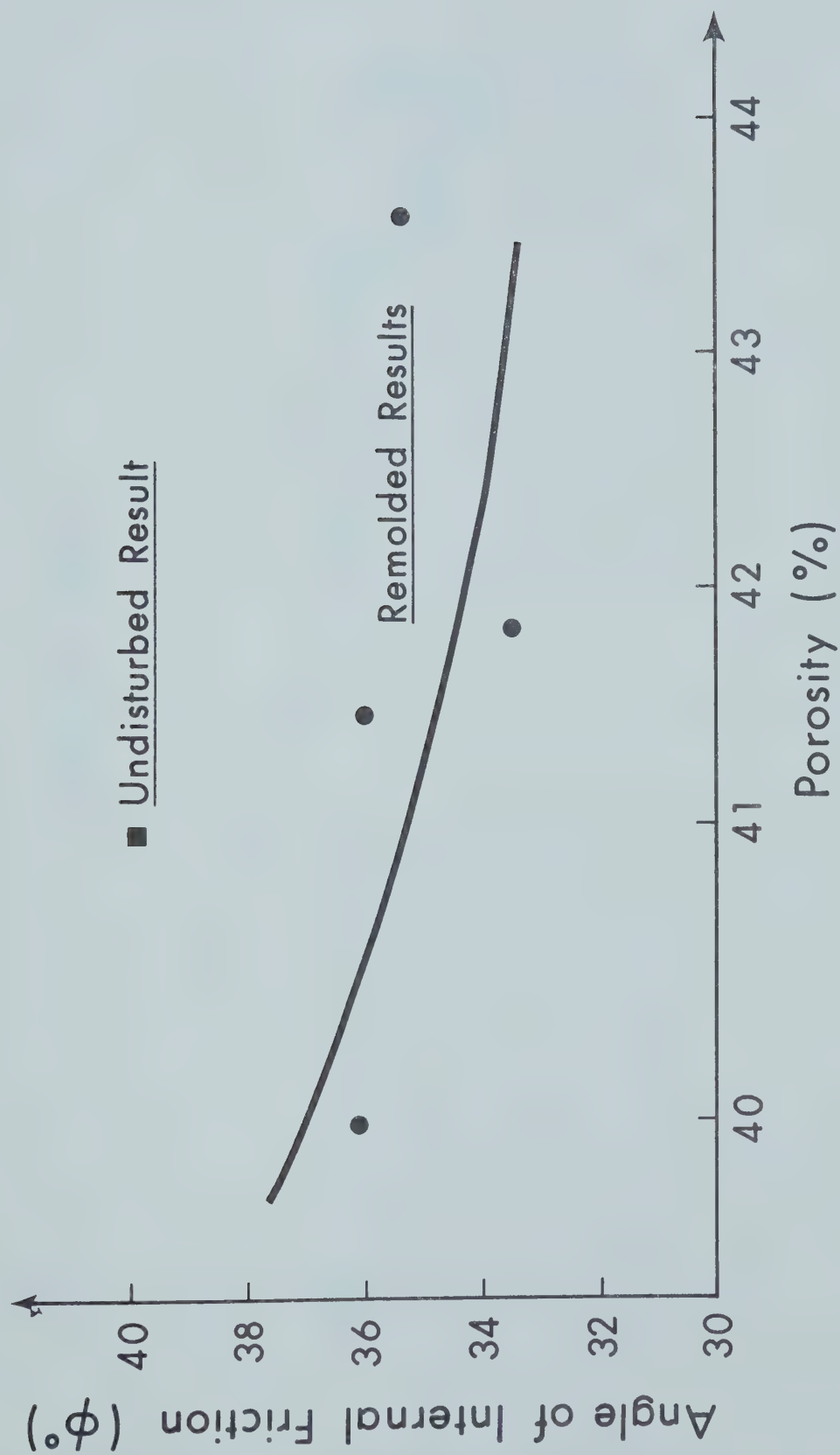


FIGURE 5.6
REMOLDED RESULTS (POROSITY vs. ϕ')

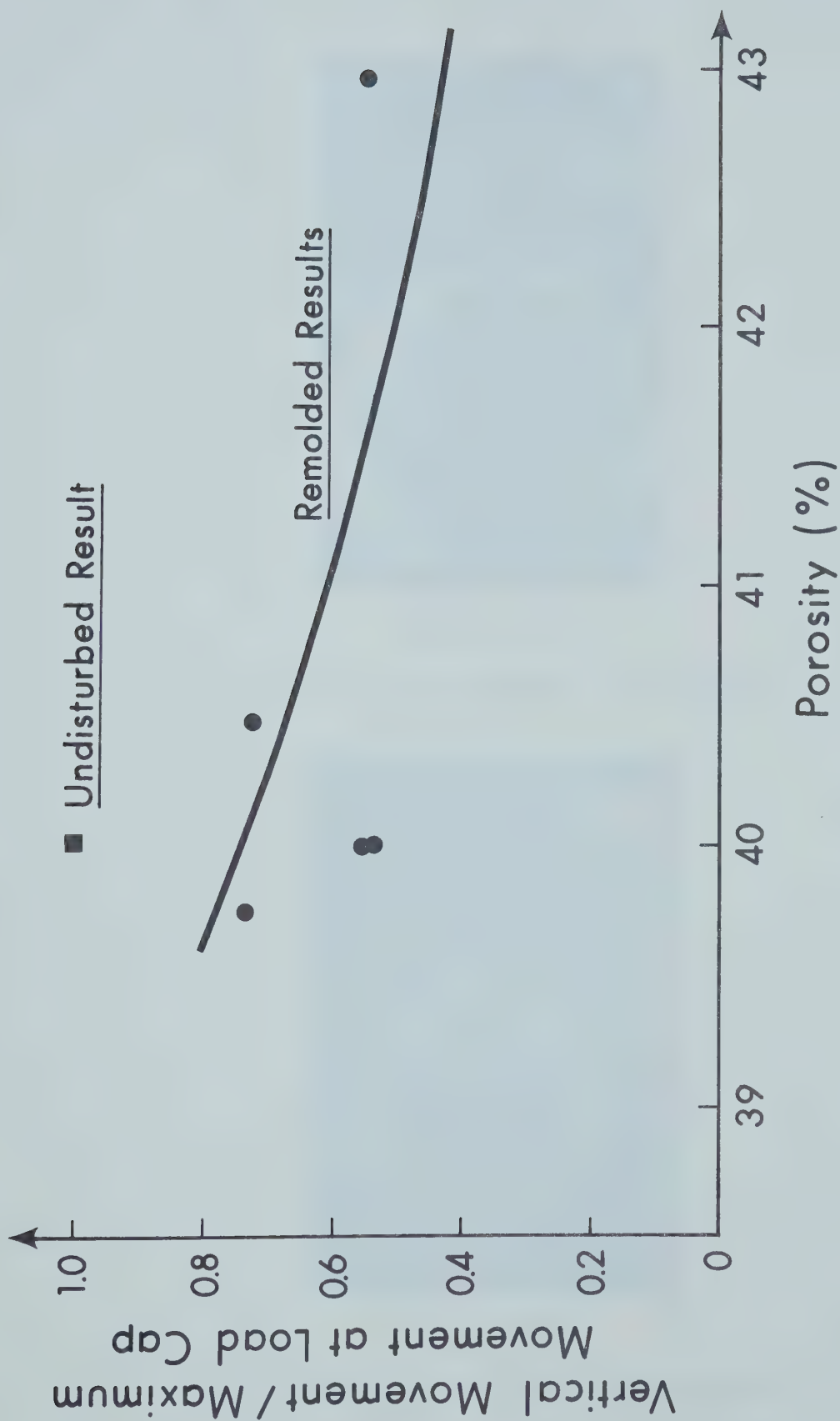


FIGURE 5.7

UNDISTURBED/REMOLDED VERTICAL MOVEMENT vs. POROSITY

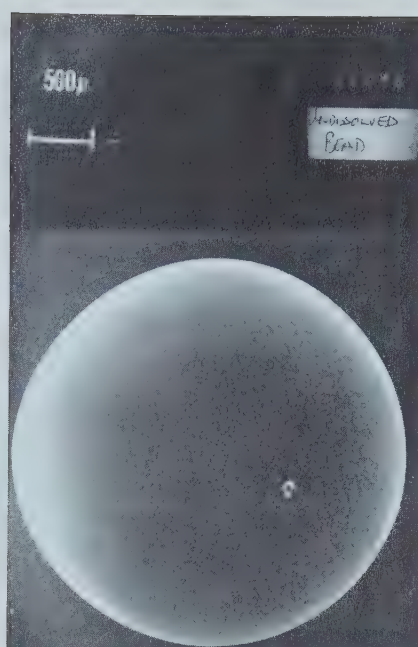


FIGURE 5.8

4mm GLASS BEAD BEFORE DISSOLUTION

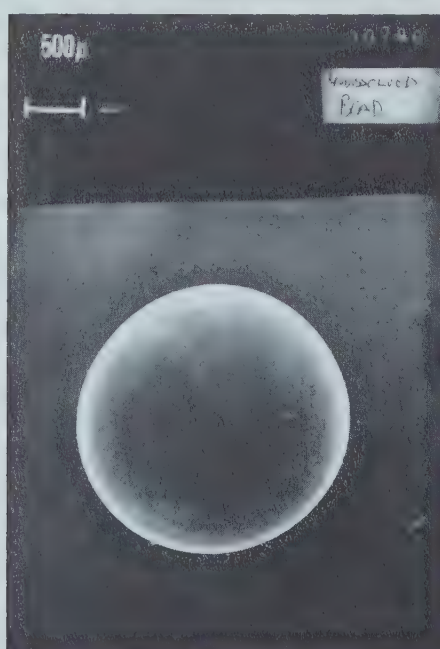


FIGURE 5.9

2-3mm GLASS BEAD BEFORE DISSOLUTION

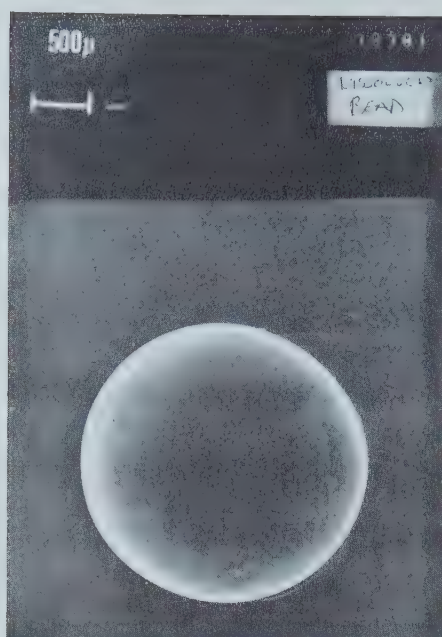


FIGURE 5.10

2-3mm GLASS BEAD AFTER DISSOLUTION

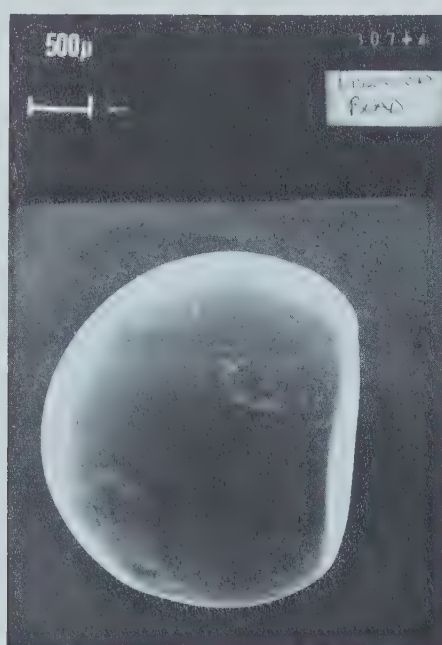


FIGURE 5.11

4mm GLASS BEAD AFTER DISSOLUTION

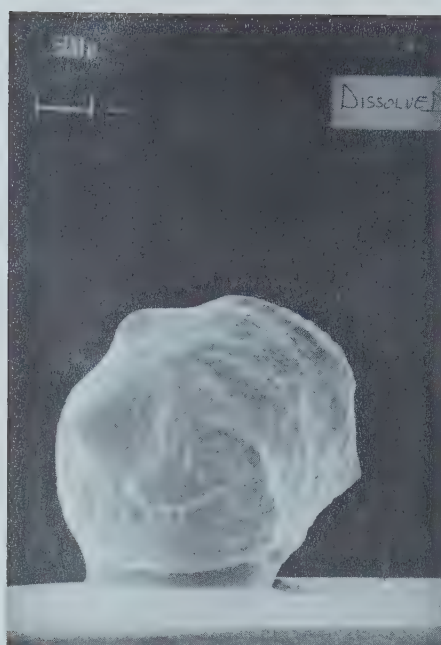


FIGURE 5.12

4mm GLASS BEAD AFTER DISSOLUTION

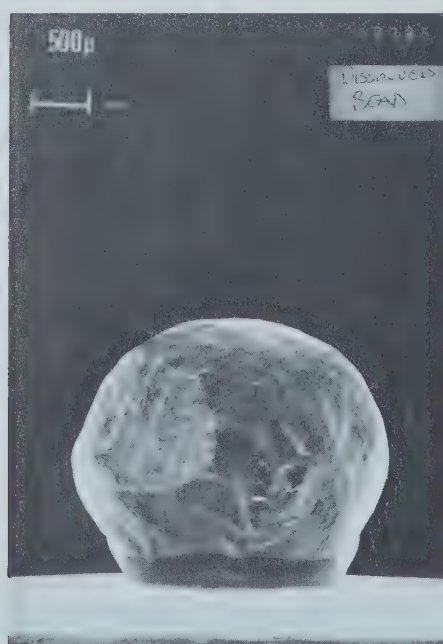


FIGURE 5.13

4mm GLASS BEAD AFTER DISSOLUTION

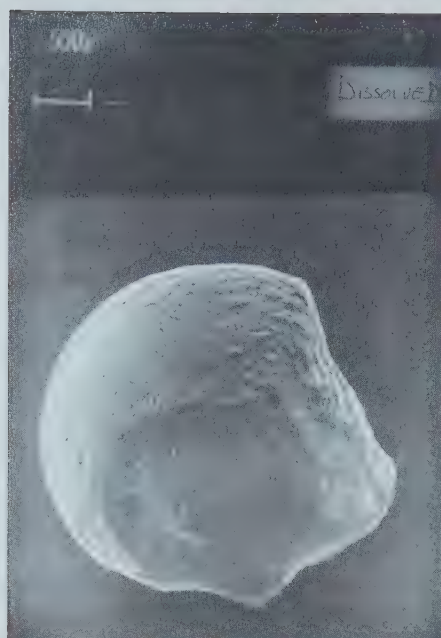


FIGURE 5.14

4mm GLASS BEAD AFTER DISSOLUTION

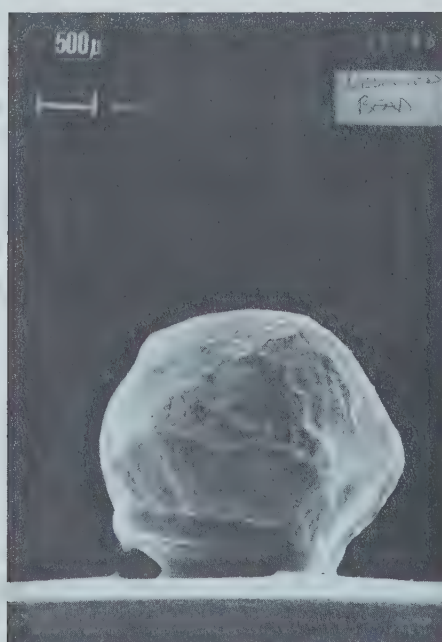


FIGURE 5.15

4mm GLASS BEAD AFTER DISSOLUTION

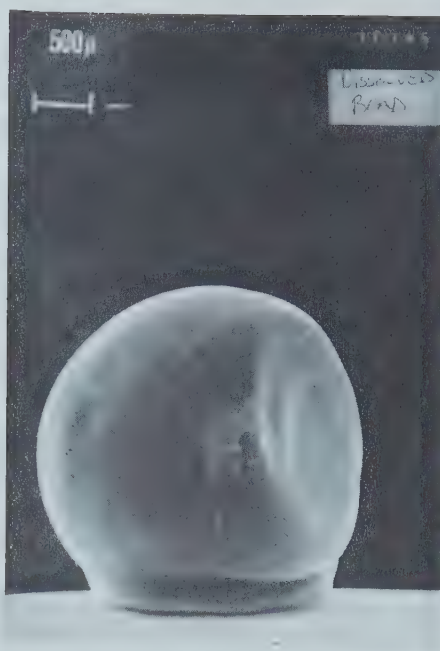


FIGURE 5.16

4mm GLASS BEAD AFTER DISSOLUTION

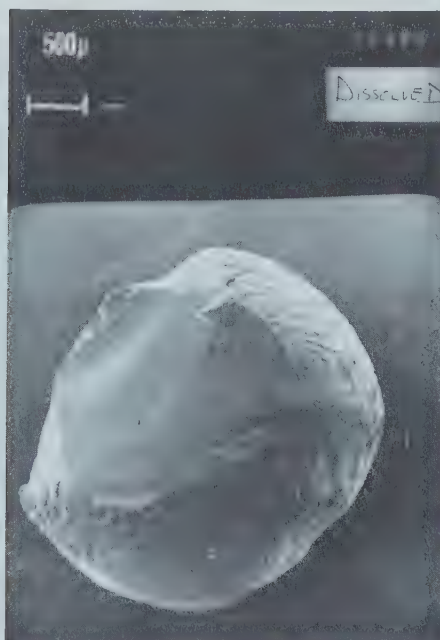


FIGURE 5.17

FRACTURE OF BEAD AFTER DISSOLUTION

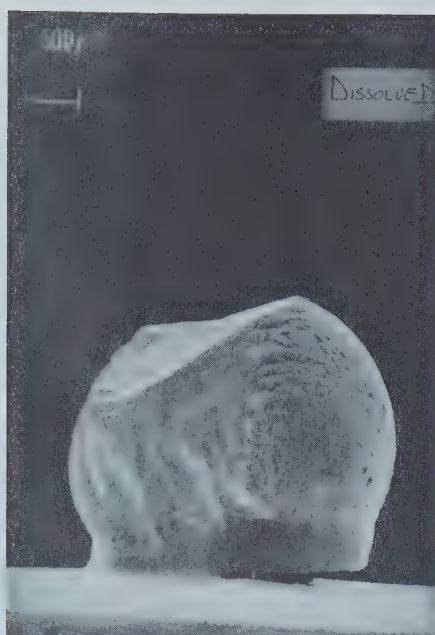


FIGURE 5.18
FRACTURE OF BEAD AFTER DISSOLUTION



FIGURE 5.19
BEAD CONTACTS BEFORE DISSOLUTION

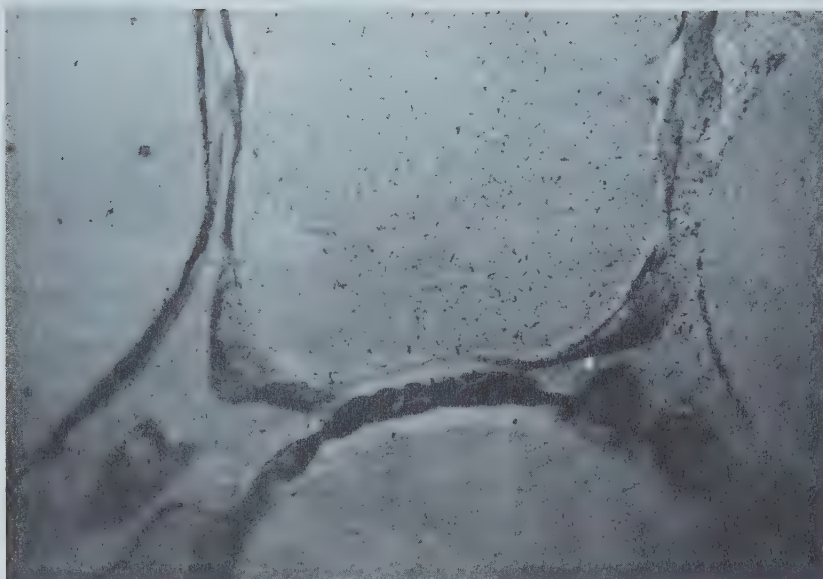


FIGURE 5.20

BEAD CONTACTS AFTER DISSOLUTION



FIGURE 5.21

BEAD CONTACTS AFTER DISSOLUTION

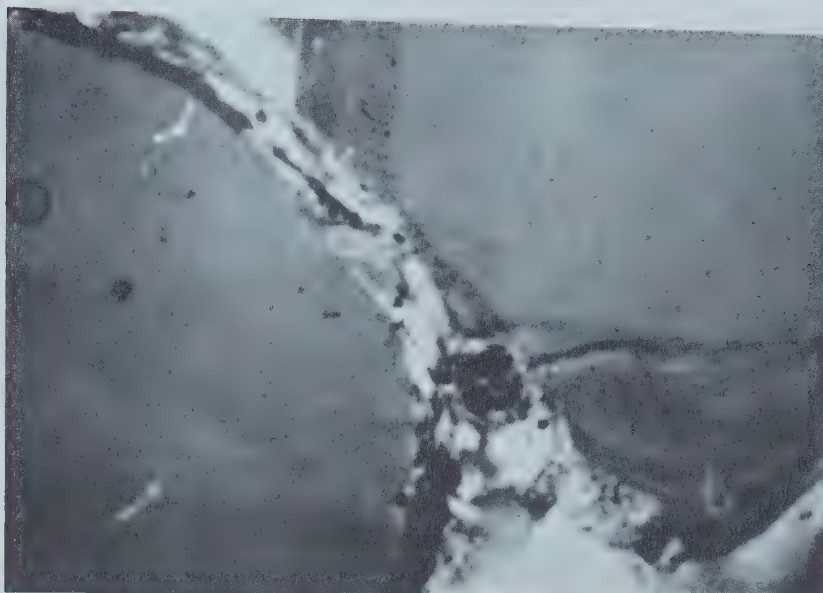


FIGURE 5.22

BEAD CONTACTS AFTER DISSOLUTION

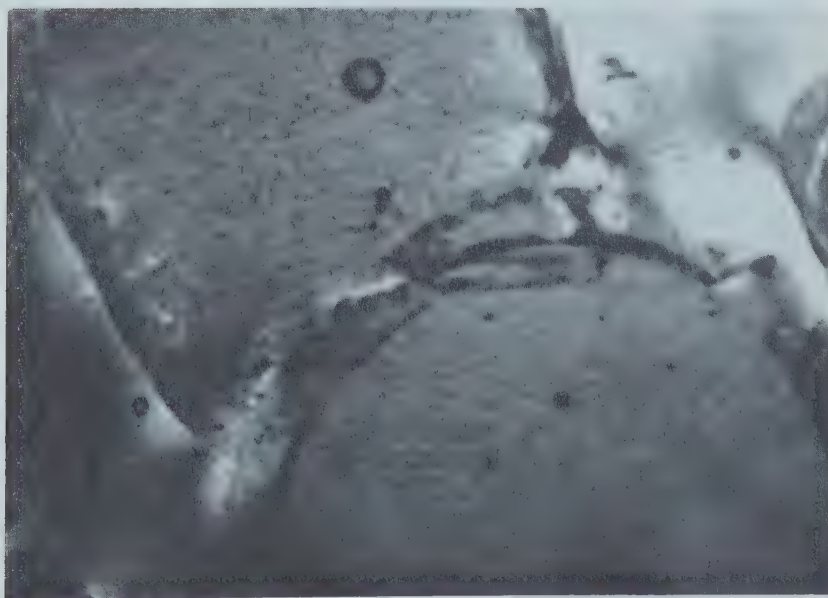


FIGURE 5.23

BEAD CONTACTS AFTER DISSOLUTION



FIGURE 5.24

STYLOLITIC CONTACT AFTER DISSOLUTION

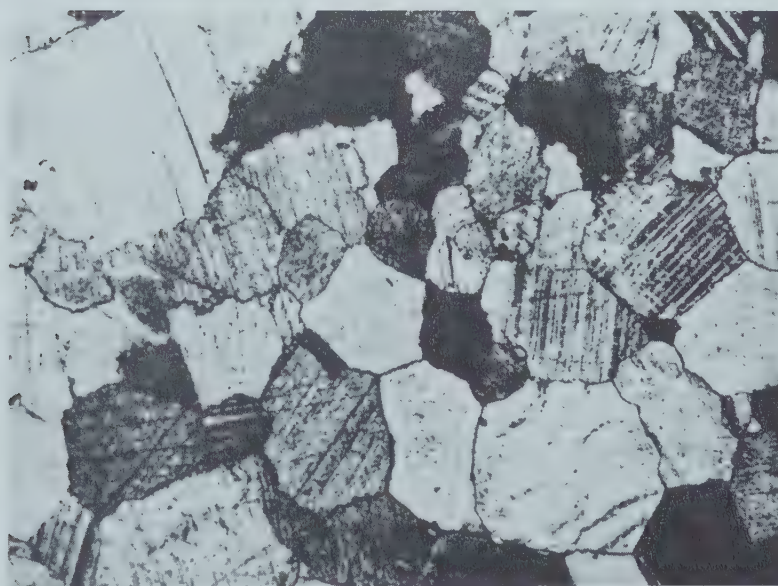


FIGURE 5.25

GRAIN CONTACTS UPON HEATING THE MARBLE

(After Rosengren and Jaeger, 1968)

CHAPTER VI

CONCLUDING REMARKS

In this thesis, an examination was made of the diagenetic processes which alter arenaceous sediments after deposition. The mechanisms responsible include pressure solution and/or authigenic overgrowths. These processes cause the sand grains to interpenetrate one another and form an interlocking structure. The absence of cement is pronounced.

Using glass beads and hydrofluoric acid, a model material was developed in which this structure was simulated. Shear strength parameters after dissolution were comparable to those obtained from the McMurray Sandstone. These are seen to differ considerably from strength parameters usually assumed for conventional granular materials.

Relating to the properties of the McMurray Sandstone, two points of considerable importance emerge :

1. Strength parameters are properly designated by a curved Mohr failure envelope.
2. For a diagenetically altered sand, recourse to index testing as a measure of strength is impracticable.

The characteristics of the McMurray Sandstone are very similar to that of the St. Peter Sandstone, another

friable sandstone. It is suggested that friable sandstones in general have characteristics which differ considerably from the classical granular material.

The characteristics of the McMurray Sandstone are also very similar to that of a randomly jointed rock. Therefore, with regard to evaluation of strength parameters in the latter, a curved Mohr failure envelope would seem more appropriate.

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